# THE NICKEL BULLETIN

## A SUMMARY OF CURRENT INFORMATION ON NICKEL

AUGUST - SEPTEMBER, 1953


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# ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

## GENERAL

### Nickel Alloy Coinage

W. A. C. NEWMAN: 'British Coinage.' *Published by Royal Inst. Chemistry*, London, 1953.

This Monograph gives an account of the history and technology of British coinage. Coining operations are described in detail, and the importance of design in relation to flow of metal is discussed. Metallurgical aspects of the subject are given full consideration, and methods of assaying and safeguards used for preservation of required coinage standards are described. A section of the monograph is devoted to discussion of alloys used for coins of various denominations, including consideration of the changes in composition which have been introduced during recent years.

*See also*—

H. G. STRIDE: 'The Royal Mint: An Outline History.' Published, for the Deputy Master and Comptroller of the Royal Mint, by Her Majesty's Stationery Office, 1953; 24 pp.

This publication gives a concise account of the history and present activities of the Royal Mint. The author deals with establishment and multiplication of mints before the Norman Conquest; the concentration of coinage in the Tower Mint; the introduction of machinery to replace the primitive method of coining money with the hammer; the present duties and work of the Royal Mint; the process of coining; the new coinage of Queen Elizabeth II, and some notes on the coins in current use.

### Powder-Metallurgy Symposium

E. BENESOVSKY (Editor): 'Plansee Proceedings, 1952. Papers presented at the First Plansee Seminar 'De Re Metallica,' 1952.'

Published by Metallwerk Plansee, G.m.b.H., Reutte/Tyrol, 1953; 316 pp.

The papers and discussion in this volume are presented in three sections, dealing, respectively, with—

General and Physical Metallurgy and Physics of Solids.
Problems of Powder Metallurgy in General.
Cemented Carbides and Other Hard Metals.

Many of the papers have direct or indirect bearing on the powder-metallurgy of nickel and nickel alloys. Among those which refer specifically to nickel-containing materials are the following:—

W. SEITH: 'Diffusion in Solid Metals,' pp. 65-80.
(Including diffusion data on nickel/iron and nickel/copper, and diffusion of carbon into nickel-iron alloy.)

G. J. COMSTOCK and F. H. CLARK: 'The Development of High-Strength, Heat-Treatable Products from Alloy Powders,' pp. 123-39.
(Including information on powder-metallurgy production of parts from low-nickel alloy steels, and austenitic stainless steel.)

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H. H. HAUSNER: 'The Effect of Lattice Changes on the Sintering Process,' pp. 146-56.
(Contains report of experiments designed to determine the influence of heat-treatment in hydrogen, and of compacting pressure, on powder compacts prepared from carbonyl nickel. Special study of electrical resistivity as a function of treatment.)

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W. J. KROLL: 'The Production of Metal Powders by Fusion Electrolysis,' pp. 160-70.
(Includes reference to production of nickel and nickel-chromium alloy powders.)

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G. TIRZAU: 'Contribution to the Study of the Physical Analysis of the Sintering Process,' pp. 188-98.
(The use of electrical conductivity and of thermo-electric potential as criteria of changes taking place during sintering is illustrated by data on the following groups: copper/nickel, copper/iron, tungsten carbide/cobalt, iron/nickel/aluminium.)

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R. EDWARDS AND T. RAINES: 'The Solid Solubilities of Some Stable Carbides in Cobalt, Nickel and Iron at 1250°C.,' pp. 232-43.
(Solubility in the solid state, at 1250°C., of (1) carbides of tungsten, titanium, tantalum, molybdenum, vanadium and chromium, and (2) titanium carbide-tungsten carbide mixed crystals, in cobalt, nickel, and iron, was determined by metallographic and X-ray methods.)

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R. KIESSLING: 'Investigations on Ternary Systems $Me_1$-$Me_2$-B, and a Discussion of the Relative Strength of the Bond Transition Metal-Boron,' pp. 297-302.
(The systems investigated included manganese-nickel-boron.)

### Temperature-Sensitive Magnetic Nickel Alloys

See abstract on p. 139.

# NICKEL

## Recent Developments in Mining of Nickel-containing and Other Ores

*The Engineering and Mining Journal* of July 1953 contains a survey of important developments which have recently taken place in the mining world. It is pointed out that the present time is 'Mining's Greatest Era', and that there has never been a time when so great an investment was being made, all over the world, in new mining enterprises and in expansion of older ones. Emphasis is laid on the opportunity thus offered for enterprise, in both research and development, among the younger generation of mining engineers and metallurgists.

Introductory articles review (1) the probable calls which will be made within the next decade for additional metal supplies to meet industrial development, and the resultant need for expansion in mining; (2) properties of some 'new' metals which have recently assumed importance in the industrial world, e.g., titanium, uranium.

Later sections of the symposium relate to mining developments concerned with individual metals:

*Iron Ore:* major projects which have been undertaken to meet increasing demand for steel, with some details of individual plants in various parts of the world.

*Copper:* a list of new projects operating, in construction, or planned, with notes on some of the more important workings and plants.

*Aluminium:* statistical data on recent growth in world supply of aluminium, and plans being made by various companies for still further development.

*Lead and Zinc:* increase in use of the metals resulting from research and development work; survey of main sources of supply; some expansion projects.

*Uranium:* reasons for the recent world-wide boom in this metal; producers in various countries; some of the main uses in connection with development of applications for atomic power.

*Titanium:* defence requirements, with special reference to aircraft; production methods and sources of supply.

*Nickel-Cobalt:*

Important developments in the Sudbury basin, including large expansion by International Nickel Company; Falconbridge activities; other companies operating in this area.

The Rhodesian cobalt-producing plant operated by Rhokana Corporation.

Developments at the Nicaro nickel plant in Cuba, operated for the U.S. Government by Nickel Processing Corporation; prospects from a nickel orebody recently reported, by Freeport Sulphur Company, at Moa Bay, Cuba.

Extraction process developed by Sherritt Gordon Mines, Ltd., for treating ores from their Lynn Lake holding in Northern Manitoba and prospects for working of a mine in British Columbia, using the same process.

Projected development of orebody near Riddle, Oregon, using an electric smelting process developed in France.

A key to the map of new mineral projects in Canada (p. 226) shows the position of seven areas in which extraction of nickel is in progress.

Later sections of the review deal with development of small mines in U.S.A.: Government assistance; problems facing the small miner; and the current position and future development of mining of non-metallic materials (asbestos, sulphur, phosphates, etc.).

## Iron-Ore Recovery from Nickel Ores

'Iron Ore Recovery from Nickel Ores.' *Economist*, 1953, Sept. 26, p. 880; see also *Metal Industry*, 1953 vol. 83, Sept. 25, p. 265.

The International Nickel Company of Canada, Ltd. announces that a by-product iron ore is to be produced from nickel ores mined in the Sudbury district of Ontario. Construction of new plant in the Copper Cliff area, which is already under way, will ultimately result in a yield of about 1,000,000 tons of iron ore per year, in addition to the increased nickel output which the Company has undertaken to supply. Initially the plant will treat 1,000 tons per day of nickel-bearing pyrrhotite removed from ore in the early stages of processing at Copper Cliff. The iron ore will be higher in grade than any now produced in quantity in North America and is likely to be in wide demand for direct use in open-hearth and electric-furnace steel production in Canada and U.S.A.

The new process, with its yield of iron ore, makes possible treatment of low-grade ores which were hitherto considered uneconomic as a source of nickel, and in many respects the recovery of iron ore as a by-product is considered to be one of the outstanding advances in the metallurgy of nickel extraction. In other plants, throughout the world, nickel production is based on processes in which the iron content of the ore is rejected in slag or in tailings, and only a minor portion of the iron content of the ore is recovered. The new method, in addition to being the first which permits recovery of high-grade iron ores, is of significance in that it opens the way for increased recovery of other elements, including sulphur, from the complex Sudbury ores.

## Hydrometallurgical Process for Extraction of Nickel from Sulphide Ores

'Hydrometallurgical Process Yields Pure Metal Powders from Sulphides.' *Jnl. of Metals*, 1953, vol. 5, June, pp. 775-9.

This article gives details of the ammonia-leach/hydrogen-reduction process to be used by Sherritt Gordon Mines, Ltd., for treatment of the nickel-cobalt sulphide concentrate obtained from the ores of the Lynn Lake area, Manitoba. The information given was supplied by Professor F. A. Forward, who has been responsible, at the University of British Columbia, for much of the research work on which the process is based. Chemical Construction Corporation has also played an important part in

connexion with engineering and design problems associated with plant required for the process. Chemical reactions involved in extraction of the nickel, cobalt, and copper are shown in chart and tabular form, and the details of the various steps are discussed. Chronological particulars are given of the development of the Sherritt Gordon process. Following extensive research in laboratory and in pilot plants, a refinery now being built at Fort Saskatchewan, Alberta, is scheduled to start large-scale operations in January 1954. Nickel and cobalt powders, as well as ammonium sulphate, will be produced.

*See also—*

'Progress in Hydrometallurgy.' *Metal Industry*, 1953, vol. 82, June 26, p. 522.

This note gives a concise summary of the main features of the process to be used at the Saskatchewan refinery.

**Corrigendum**

*Nickel Bulletin*, 1953, vol. 26, June, p. 92. Abstract on 'New Unit for Sponge Nickel.' In reference, for '*Steel*' please read '*Chemical Engineering*'.

**Hall Effect in Cobalt-Nickel Alloys and in Nickel**

See abstract on p. 139.

**Thermal Conductivity of Nickel and Nickel Alloys: Summary**

L. SILVERMAN: 'Thermal Conductivity Data Presented for Various Metals and Alloys up to 900°C.' *Jnl. of Metals*, 1953, vol. 5, May, Sect. 1, pp. 631-2.

This paper reports results of determinations of thermal conductivity, over the range room temperature to 900°C., for some metals and alloys used in the electron-tube industry. The procedure used in the tests was a modified form of the comparative method developed by Van Dusen and Shelton, in which the conductivity of other metals is related to that of a standard. In the examinations made by Silverman lead was chosen as the primary standard because the absolute value of the thermal conductivity of lead at 0°C. is generally accepted (0·352 watts/cm$^{-1}$/deg.$^{-1}$). The compositions of the materials on which determinations were made, and their relative conductivities at the respective temperatures, are shown in tables.

The materials include Lohm Alloy (copper 93·4, nickel 6·05 per cent.), A nickel (commercial grade); Advance Metal (45/55 nickel-copper alloy); D nickel (containing 4 per cent. of manganese); Monel (nickel 66·2, copper 30 per cent., with small amounts of iron and manganese); R.C.A. alloys Nos. N 91 and N 97 (nickel-cobalt alloys); austenitic nickel-chromium stainless steels; nickel-chromium alloy (80-20 type); Hastelloy A (nickel-molybdenum-iron alloy); straight-chromium stainless steels; Inconel (nickel 78·13, chromium 13·94, iron 6·33 per cent.); nickel-iron alloys (52 and 42 per cent. nickel types); Svea high-purity iron; Kovar (iron 53·7, cobalt 17·15, nickel 28·75 per cent.); titanium.

**Oxidation of Metals at High Temperatures**

See abstract on p. 147.

**Colour and Reflecting Properties of Stainless Steels and Other Materials**

See abstract on p. 153.

**Influence of Mode of Preparation on Catalytic Properties of Nickel and Cobalt**

F. LIHL: 'Influence of Temperature of Reduction of Nickel and Cobalt Compounds on the Catalytic Properties of the Metallic Catalyst.' *Zeitsch. für Elektrochemie*, 1952, vol. 56, Oct., pp. 979-85.

The author's main finding, applicable to hydrogenation at 130°-140°C., is that nickel catalysts show fall in activity with time, irrespective of the nature of the starting material or the temperature of reduction. The higher the initial activity the more marked is the decrease. Initial activity of nickel catalysts decreases with rising temperature of reduction. For cobalt catalysts efficiency is unaffected by reduction temperature over a considerable range; decrease in activity is observed only with reduction temperatures above 500°C.

Differences found between nickel and cobalt powders are ascribed mainly to the fact that nickel is always present as a single phase, whereas cobalt, under the conditions of preparation used, generally consists of two crystal structures, one superimposed on the other.

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F. LIHL and P. ZEMSCH: 'Catalytic Properties of Nickel and Cobalt Prepared from Amalgams by Removal of Mercury by Distillation or by Decomposition in Air.' *ibid.*, pp. 985-7.

Investigation of catalysts prepared from amalgams of cobalt and of nickel showed that nickel powders thus obtained are inactive. The activity of cobalt powders made by the same method, and the time behaviour of the catalysts, depend on whether the powder has been prepared by decomposition of amalgam in air or by removal of mercury by distillation.

**Nickel and Nickel Alloys in Electronic Equipment**

K. JACKSON and R. O. JENKINS: 'Nickel in Electronics.' *Metallurgia*, 1953, vol. 47, June, pp. 277-82.

The manufacture and operation of thermionic valves impose requirements of a somewhat unusual nature upon the materials used in their construction. The various electrodes comprising the valve assembly, the pinch and lead-in wires which pass through the glass envelope, and the getter used in production and maintenance of the high vacuum, have widely differing functions, each of which calls for consideration from a metallurgical point of view. With few exceptions, nickel or a nickel alloy satisfies these diverse requirements; and for many purposes in valve construction there are no adequate alternatives.

The manufacturer of valves and cathode-ray tubes necessarily demands closer control of composition and purity than is usual for some other applications, and

**The Uses of Nickel in Valve Components**

| Use | Alloy* | Nominal Composition % |
|---|---|---|
| Indirectly heated cathodes | 'O' nickel<br>Inco '220' nickel (U.S.A.) | 99·5 min. (Ni+Co)+ controlled Mg |
| Directly heated battery-operated cathodes | 0·25% magnesium nickel | As stated |
| Directly heated battery-operated cathodes | 0·4% aluminium nickel | As stated |
| Directly heated cathodes (50 mA. range) | N.93 | 2 W, 1 Al, 0·2 C, balance Ni. |
| Directly heated cathodes (50 mA. range) | N.100 | 2 W, 1 Al, 0·2 C, 0·2 Mg, balance Ni |
| Mercury rectifier cathodes | Cobanic | 40 Co, 0·2 C, 0·2 Si, balance Ni |
| Anode support wires | 'A' and 'G.F.A.' nickel | 99·0 min. (Ni+Co) |
| Grid winding wires and grid side rods | 5% manganese nickel | 4·5-5·5 Mn, balance Ni |
| C.R.T. gun electrodes and deflection plates | Nickel-copper | 45 Ni, 55 Cu |
| Valve springs | Nimonic 90 | 15-21 Co, 18-21 Cr, 5 max. Fe, balance Ni |
| Glass-to-metal seals— | | |
| Soft glass | 'Dumet' (copper-clad) | 41-43 Ni, balance Fe alloy, clad with Cu |
| Soft glass | Nickel-iron | 49-51 Ni, balance Fe |
| Soft glass | Nilo 475, No. 4 alloy, etc. | 42-47 Ni, 4-6 Cr, balance Fe |
| Borosilicate glass | Nilo K, Kovar, Nicosel, etc. | 28-30 Ni, 16-18 Co, balance Fe |

* Some of these materials are described by trade marks.

this paper has been written, by a valve engineer and a metallurgist, to explain some of the metallurgical problems involved and the materials which have been developed for various components used in the electronics industry.

General requirements for metals used in valve construction are outlined and particular attention is called to the importance of the gases present in the metals and outgassing treatments which are applied to nickel. The application of nickel and nickel alloys to valve components is then discussed at length, on the basis of the particular parts in which they find application, and the qualities and properties required for successful functioning in the respective components. The Table above shows the type of materials so considered.

Oxide-coated cathodes are dealt with in two classes: those which are indirectly heated by a heater electrically insulated from the cathode proper, and the directly heated type, in which a current is passed through the cathode to raise it to the operating temperature. The working mechanism of each type is described, and the composition limits and properties of the materials most suitable for such parts are discussed. Anode materials are then reviewed, and the final section of the survey is concerned with alloys used in glass-to-metal seals, in which a primary requirement is closely controlled thermal-expansion characteristics, matched to the various types of glass with which they are to be sealed. Particular mention is made of the nickel-iron and nickel-cobalt-iron alloys in this connexion.

**Nickel-base Oxide Cathodes**

A. M. BOUNDS and P. N. HAMBLETON: 'The Nickel-base Indirectly Heated Oxide Cathode.'
*Electrical Engineering*, 1953, vol. 72, June, pp. 536-40.

The barium-oxide indirectly heated oxide cathode came into wide use in 1927 and has since then virtually dominated the field of ordinary receiving tubes and also of many of the special types. The present authors describe the essential components and characteristics of the complex system which comprises the cathode, and point out the many factors involved in cathode performance. The discussion is concerned principally with the nickel cathode alloys used as base: detailed consideration is given to the composition of these materials, as affecting the behaviour of the cathode in service.

The chemical reduction of earth-metal oxides by elements contained in the nickel sleeve is important. With few exceptions, all cathode nickels contain controlled amounts of one or more of the elements magnesium, silicon, titanium, aluminium, carbon, and tungsten, as chemical reducing agents for the alkaline-earth-metal oxide coating. Each of these elements, singly and in various combinations, affects the performance characteristics in different ways; the choice of an alloy of optimum composition is therefore of major importance. This paper contains a critical summary of the influence of the elements mentioned above, and also includes brief consideration of some mechanical variables which may affect the life and behaviour of cathode assemblies. A table gives compositions of commercial nickel-cathode materials in

current use in U.S.A. These are classified, in accordance with the terminology adopted by the American Society for Testing Materials, in three groups designated, respectively, 'active', 'normal', and 'passive' alloys. Such designation, although not exactly definitive as related to performance characteristics, serves as a broad classification on the basis of rapidity of activation and level of d.c. emission during life.

### Nickel Equipment in Salt-Producing Plant

A. W. BAMFORTH: 'Modern Plant for Salt Production.' *Times Rev. Industry*, 1953, May, pp. 28-9.

This article describes the operating principles of the Krystal process for production of sodium chloride, which, although developed in 1920, was not until recently applied on an industrial scale. A description is given of a triple-effect Krystal sodium-chloride evaporator installation designed and fabricated by Power-Gas Corporation, providing for production, from raw brine, of 300 tons per day of 99·9 per cent. sodium chloride, required for manufacture of chlorine and caustic soda.

The first-effect evaporator of this plant, which operates at a pressure near atmospheric, is entirely in solid nickel, while the second- and third-effect evaporators are composite constructions of high-grade cast iron and nickel. The design of the nickel shells follows the general requirements of B.S. 1500 for medium-duty steel vessels. The evaporators, which are 14 ft. 6 in. internal diameter and over 45 ft. in overall height, are believed to be among the largest solid-nickel vessels ever constructed in Great Britain.

Large-capacity circulating pumps of Ni-Resist (high-nickel-alloy cast iron) are also a feature of the installation described.

### Nickel-Alkaline Batteries for U.S. Aircraft

'French Battery Challenges U.S. Types.'
*Amer. Aviation*, 1953, vol. 17, June 22, pp. 61-2.

The article emphasizes the value of nickel-cadmium batteries, on the ground of long life, greater reliability, reduced maintenance, and simplified installation. The French firm Société des Accumulateurs Fixes et de Traction (S.A.F.T.) is under contract to deliver (by January 1954) 2,000 nickel-cadmium batteries to the U.S. Bureau of Aeronautics, and it is anticipated that within about two years nickel-cadmium batteries may entirely replace lead-acid models in all U.S. military aircraft. Manufacture of the French type of battery in U.S.A. is envisaged.

### Powder-Metallurgy Symposium

See abstract on p. 126.

### Enamelling of Sheet Nickel

J. H. HEALEY and A. I. ANDREWS: 'A Study of the Enamelling of Sheet Nickel.'
*Bull. Amer. Ceramic Soc.*, 1953, vol. 32, Apr., p. 119.

Enamelling of sheet nickel has presented difficulty, due to extensive formation of bubbles and lack of adherence. This note reports investigation of changes occurring at the enamel-nickel interface, and the effects of various heat-treatments of the sheet nickel prior to enamelling.

It is concluded that the gases in the bubbles formed are not reducing, since no metallic particles form on the surface of the bubbles in enamels containing cobalt, nickel or copper oxides. It is believed that oxidation of the sheet nickel before enamelling accounts for formation of sufficient quantities of carbon dioxide to make the carbon gases evolved neutral or slightly oxidizing.

Heat-treatment of the sheet in air, nitrogen, or argon, prior to enamelling, was not found to lessen formation of bubbles at the enamel-nickel interface, but treatment in annealing gas containing nitrogen 85, hydrogen 15, per cent., had a markedly beneficial effect. Sheet nickel treated in this atmosphere for two hours at 1800°F. (982°C.) was enamelled with formation of only a few scattered bubbles. Treatment in the same gas at lower temperatures was less effective.

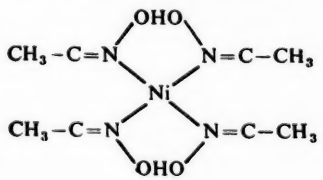
### Structure of Nickel Dimethylglyoxime

L. E. GODYCKI and R. E. RUNDLE: 'The Structure of Nickel Dimethylglyoxime.' *Acta Crystallographica*, 1953, vol. 6, June 10, pp. 487-94.

The work reported in this paper led to the following conclusions:

'Nickel dimethylglyoxime is orthorhombic, $a_o = 16 \cdot 68$, $b_o = 10 \cdot 44$, $c_o = 6 \cdot 49$ Å, $Z = 4$, space group *Ibam*. The molecules are required to have symmetry $2/m$, and within the limits of error of this investigation, have the higher symmetry *mmm*.

'The expected structure was confirmed: see below:—




'The intramolecular O.H.O. bonds are the shortest yet observed, 2·44 Å. An explanation is advanced to explain the difference in solubilities of copper and nickel dimethylglyoximes in water, and the unusually small pleochroism.'

Crystallographic data are included for the corresponding palladium compound, and for nickel cyclohexanedionedioxime.

### Spot Test for Detection of Copper, Cobalt and Nickel

H. F. SCHAEFFER: 'A Multiple Spot Test for Copper, Cobalt and Nickel.' *Chemist Analyst*, 1952, vol. 41, Sept., pp. 57, 59.

The method described is based on the use of isoquinoline-ammonium thiocyanate. (The author had previously reported that this reagent could be used in the microchemical detection of several cations, through separation of the resulting metallo-organic compounds in the form of microscopic crystals.) An

advantage of the procedure is that none of the three metal ions concerned interferes with the other two.

The following solutions are required:—

*Isoquinoline Hydrochloride.* A 0·4 molar solution of isoquinoline hydrochloride, prepared by mixing 12·9 g. (0·1 mole) of isoquinoline with 100 ml. of 1-*N* hydrochloric acid and diluting to 250 ml.

*Ammonium Thiocyanate.* A 0·4 molar solution of ammonium thiocyanate, prepared by dissolving 3·0 g. of ammonium thiocyanate in sufficient water to make 100 ml.

*Dimethylglyoxime.* A solution of dimethylglyoxime in alcohol or acetone.

*Ammonia.* A bottle of concentrated ammonium hydroxide.

*Spot Test*

The test papers are prepared from 7-cm. circles of qualitative filter paper, by placing a drop of the isoquinoline hydrochloride solution in the centre of each circle. After a brief interval a drop of the ammonium thiocyanate solution is added to the isoquinoline hydrochloride spot. (It is preferable to allow most of the moisture to evaporate from the paper before use.)

The sample to be tested may contain the metal ions with chloride, acetate, nitrate or sulphate, but excess acid should be avoided. In performing the test, one drop of the sample is applied to the centre of the reagent spot. The presence of cupric ion is revealed by a light yellow-green coloration; cobaltous ion shows blue. In the presence of both these ions, the green colour develops in the centre, while an irregular blue corona forms outside this area. (The effect is illustrated in the original.) The next step is to render the spot alkaline, by exposing over the mouth of a bottle of ammonium hydroxide for several seconds. Finally, a drop of dimethylglyoxime solution is added to the spot, and the presence of nickel is indicated by formation of the usual pink or red colour.

Well-defined tests are obtained at a metal-ion dilution of 1 part in 3,000, but it is considered that this does not represent the lower limit. Zinc and cadmium yield a white precipitate with the isoquinoline hydrochloride, and therefore do not ordinarily interfere with the test. Excess ferric iron masks the effects, by developing a deep red colour, but slight traces of iron do not interfere.

### Determination of Nickel in Cobalt Oxide

J. H. MCCLURE and R. E. KITSON: 'Spectrochemical Determination of Impurities in Cobalt Oxide.' *Analytical Chemistry*, 1953, vol. 25, June, pp. 867-8.

Although conventional methods of analysis are available for estimation of various impurities liable to occur in cobalt oxide, many of these procedures are time-consuming, and some of them lack precision. This paper describes a direct spectrographic method developed for determination of calcium, barium, copper, silica, iron, manganese, chromium, aluminium, and nickel, in cobaltic oxide. The procedure is rapid, and, in general, 0·001-0·1 per cent. of the impurities can be estimated with an accuracy and precision of about ±20 per cent. relative. The authors give details of line pairs and their usable ranges: they also describe a solution method for preparation of standard samples.

For several of the elements mentioned, an independent method of analysis was used to check the accuracy of the spectrographical results. *Inter alia*, comparison of nickel determinations by this method and by the polarographic procedure developed by LINGANE and KERLINGER (*ibid.*, 1944, vol. 13, p. 77) showed good agreement.

## ELECTRODEPOSITION AND OTHER COATING METHODS

### Cleaning of Metals for Electroplating

H. B. LINFORD and E. B. SAUBESTRE: 'Cleaning and Preparation of Metals for Electroplating. VI. Sensitivity of Degreasing Evaluation Tests.' *Plating*, 1953, vol. 40, May, pp. 489-96; June, pp. 633-4, 639-45.

This report is a continuation of the series being issued under the auspices of A.E.S. Research Project No. 12. The work covered in the present section was comparison of the sensitivities of various degreasing evaluation tests when oil residues are present as uniform films (as contrasted with the oil-distribution in the 'Sequential Testing' experiments, previously reported, when the oil on the surface was in the form of discrete droplets). The authors conclude that the sensitivity of degreasing evaluation tests is:—

atomizer > spray - pattern (water - break) > fluorescent dye > potassium-ferricyanide paper and copper-sulphate dip.

### Electrodeposition of Nickel: Technique, Properties of Deposits, etc.

Brief abstracts have been published of papers read and discussed at the 1953 Convention of the American Electroplaters' Society, held in June in Philadelphia, Pa.: see *Plating*, 1953, vol. 40, June, pp. 657-61. The notes below give some indication of the scope of some of the communications. Further reference will be made, in The Nickel Bulletin, to the papers as they appear in full in the Proceedings of the Society, or elsewhere.

W. L. PINNER: 'A Further Study on the Effect of Abrasive Metal Polishing on the Character of Nickel Plate,' p. 659.

By means of photomicrographs, the character of steel surfaces produced by various abrasive polishing procedures is shown, and the effect of such surfaces on the quality of the nickel electrodeposited upon them is discussed, with reference to smoothness, buffability, and corrosion-resistance. The beneficial effect of certain electrochemical treatments, in removing fragmented particles prior to plating, is considered.

E. J. SERFASS: 'The Analysis of Electroplating Solutions for Major Constituents,' p. 660.

This report covers part of the work in progress under A.E.S. Research Project No. 2. An evaluation is made of procedures for estimation of the major constituents

of common nickel-plating baths. The information given covers simple, rapid, and yet precise procedures for determination of nickel, boric acid, sulphate, chloride, etc., and, in order to render the report more practically useful to platers, cost figures for various procedures are included.

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D. T. EWING, J. K. WERNER, A. A. BROUWER, and C. J. OWEN: 'The Effect of Chromium in Nickel Plating Solutions, and Methods for its Removal.' pp. 660-1.

This paper is one of a series on the effects of impurities in nickel-plating solutions, and methods of purification. Changes in the physical properties of nickel electrodeposits (adhesion, ductility, hardness, etc.), as influenced by the presence of chromium in the electrolyte, are considered, and the effect of chromium on covering power and on salt-spray corrosion-resistance is also discussed. Details are given of procedure for removal of chromium by reduction followed by precipitation, and the differing effects of trivalent and hexavalent chromium are described.

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A. E. R. WESTMAN: 'A Metallographic Study of Some Steels used for Nickel Plating.' p. 661.

A review is made of the various types of steel (killed, semi-killed, and rimming) which may be encountered by the plater: microstructures characteristic of the respective grades of steel are illustrated. Results of a study of surface finishes having varying Profilometer readings are given, and the effects, on the electrodeposited coatings, of different modes of surface preparation are shown.

**Barium-Peroxide Additions to Nickel-Plating Baths**

J. M. ZANDER: 'The Use of Barium Peroxide for Extending the Life of a Nickel Bath.' *Better Enameling*, 1952, vol. 23, Aug., pp. 6-7, 32.

In the March 1952 issue of this journal suggestions were made as to the possibility of using barium-peroxide additions for control of nickel baths and conservation of nickel salts in preparation of work for enamelling. This method was recommended as providing a practical and chemically sound method of regulating pH and ferrous sulphate content, and for preventing formation of sodium sulphate resulting from the chemical reaction which takes place when caustic or soda ash is used.

Tentative recommendations were then made as to procedure, and three enamelling plants have given the method large-scale test. Their experience is reported in the present article, and, on the basis of the tests, modified procedure is proposed, by which maximum benefit may be obtained without necessitating any radical changes in present practice.

**Roughness in Electrodeposited Nickel**

C. J. SWANSON: 'Roughness in Bright Nickel Electrodeposits.' *Trans. Inst. Metal Finishing*, 1953, vol. 29; *Bull. Inst. Metal Finishing*, 1953, vol. 3, pp. 91-112.

Among the conditions which encourage formation of discrete nodular growths, resulting in roughness, are the presence of insoluble or undissolved material in the bath, excessive current density, incorrect temperature, porous or scratched basis metal, and irregular crystal growth of the deposit. These general conditions of bath operation have been recognized as promoting defects of this type, and are consistently avoided in reputable plating establishments. Roughness of commercial electrodeposits, on a scale such as to cause serious concern, is at present rare.

The present report (on an investigation made by the British Non-Ferrous Metals Research Association) gives an account of a form of roughness which had been observed from time to time on commercial bright nickel electrodeposits, and for the occurrence of which operating conditions appeared to provide no adequate cause. The investigation comprised (1) an examination of nodules on the articles, and of anodes and samples of electrolyte taken at times when nodular deposits were being produced, and (2) laboratory attempts to cause nodule formation by the addition of various particles to the electrolyte.

The conclusions drawn from the examination (extensive details of which are given in the paper) are summarized below:—

Nodules of a smooth contoured type are formed upon conducting particles lying on the surface, and at scratches in the surface, a finding which indicates that careful preparation of the basis metal is the key to avoidance of such defects, but since nodules of this type have a smooth and well-rounded surface they are not likely to prove objectionable unless they become numerous. A rough-contoured nodule is, however, much more clearly evident and constitutes a greater threat to general finish. The origin of this type of defect did not become apparent during the examination reported, but it was found possible to suggest a mode of formation, and typical examples of the defect are discussed in relation to the observations made. It is believed that growth of this type takes place on irregularly shaped nickel particles produced by faulty corrosion of the anode: it was found possible in laboratory tests to cause formation of similar particles by working an anode at low current density or in a solution having a very low chloride content.

Since the examination of the commercial specimens of plating, anodes, and electrolyte had established that the main cause of roughness is solid impurities in the electrolyte, further laboratory work was planned to determine the specific effect of individual impurities in the electrolyte, on the quality of the deposit. The particles introduced into the solution were graphite in water, black and green nickel oxides, carbonyl iron powder and carbonyl nickel powder. The degree of contamination was purposely higher than that which would be encountered in practice. It was found that carbon particles have a much greater effect on the roughness of the plating than any of the other impurities studied. Metallic and oxide impurities, in most cases, caused nodules, by settling on to the cathode; faces in the vertical plane were unaffected by their presence. Particles of green nickel oxide did not always affect the surface of the plate, even when the particles were comparatively large.

### Spectrographic Analysis of Nickel-Plating Baths

M. SCALISE: 'Spectrographic Determination of Nickel and Cobalt Contents of Bright-Nickel Plating Baths.' *Metallurgia Italiana*, 1953, vol. 45, May, pp. 170-4.

Details of procedure are given for estimation of the nickel and cobalt contents of bright-plating baths, and for determination of impurities liable to occur in such solutions.

### Plating of Copper and Copper-base Alloys

AMER. SOC. TESTING MATERIALS: 'Proposed Tentative Recommended Practice for Preparation of Copper and Copper-base Alloys for Electroplating,' contained in *A.S.T.M Preprint 14*, June, 1953; pp. 19-25.

The schedule (which, it is emphasized, is issued solely as a guide, and not as standardized procedure) covers brief notes on preparation of surface; recommendations on maintenance and cleaning of solutions for plating of copper-base materials; prevention of spotting out, by ensuring sound basis material; safety precautions. Fuller details are given of preparatory cycles suitable for use on (*a*) buffed work, (*b*) bright-dipped or pickled work, (*c*) work to be barrel-plated; cleaning, tarnish- and stain-removal, neutralization; copper- and silver-striking.

An appendix to the schedule contains particulars of formulae for the following solutions which may be used for copper and copper-base alloys at various stages of preparation for plating: sulphuric-acid pickle, for removal of oxides; 'fire off' or scaling dip; dichromate pickle for removal of stain after sulphuric-acid pickling; nitric/phosphoric acid pickle, for removal of oxides; electropolishing solutions.

### Nickel+Chromium (or Rhodium) Plating of Nickel Silver

G. SCHORE: 'Some Experiences in Plating on Nickel Silver.' *Plating*, 1953, vol. 40, June, pp. 628-9.

This note reports experience in a plating shop engaged in electrodeposition of nickel+chromium and nickel+rhodium coatings on fountain-pen parts, which are finally engine-turned. The following cycle of operations is normally found to give satisfactory results on nickel silver:—

Soak-cleaning in a mild solution; water-rinsing (optional); electro-cleaning, cathodic, in a mild solution, for 30-60 seconds at 6 volts, and 180°F. (88°C.); water rinsing; anodic etching in 15 per cent. (by wt.) sulphuric-acid solution at room temperature, for 15-30 seconds, at 6 volts; water rinsing; nickel plating from a proprietary electrolyte, at 30 amp./sq. ft., for 4-5 minutes, pH 4·0-4·5 (colorimetric); water rinsing and drying, *or* chromium plating from proprietary bath, for 2 minutes, *or* rhodium plating in a sulphate solution, for 2 minutes.

This method was successfully used on the pen parts, with one modification, i.e., the substitution of a 5 per cent. sulphuric-acid immersion treatment for the anodic sulphuric-acid operation, to avoid etching on parts required to have a fully bright plated finish.

On one batch of material some difficulty was experienced in obtaining adherence adequate to permit of subsequent processing of the pen and pencil caps by piercing of the vent hole without peeling of the coating. Study of the problem led to the decision to activate the nickel silver prior to plating. The following schedule was found to give good results:—

Soaking in a mild alkaline cleaning solution; water rinsing; electro-cleaning, cathodic at 6 volts, for 15-30 seconds, in mild alkaline solution; water rinsing; cyanide activation in 16 oz./gall. solution of sodium cyanide, cathodic, room temperature, for 15-30 seconds at 6 volts; water rinsing; immersion in 5 per cent. by vol. sulphuric-acid solution for 10 seconds; water rinsing; nickel plating from proprietary solution, pH 3·0 (colorimetric), operated at 15 amp./sq. ft. for 2 minutes, at 130°F. (55°C.); water rinsing; chromium plating from proprietary solution; water rinsing; drying.

The writer emphasizes that observance of generally accepted good plating practice is essential for success in any cycle of operations: he particularly stresses the importance of adequate rinsing.

### Electroplating of Beryllium

J. G. BEACH and C. L. FAUST: 'Electroplating on Beryllium.' *Jnl. Electrochemical Soc.*, 1953, vol. 100, June, pp. 276-9.

This paper describes in detail methods developed for electroplating of beryllium, (1) by a direct method, or (2) after zinc-immersion treatment.

In both cases preliminary preparation includes solvent degreasing, cathodic cleaning in a caustic solution, rinsing, chemical polishing in a solution containing (%) $H_2SO_4$ 5, $H_3PO_4$ 75, $CrO_3$ 7, balance water, operated at 120°F. (50°C.); rinsing; outgassing; chemical activation, by pickling in 10 per cent. by vol. of conc. $H_2SO_4$ at about 80°F. (25°C.); final rinsing.

Following this cycle, beryllium may be directly plated by the procedure shown below:—

**I. Direct Method**

(*a*) *Anodic pickling* — % by vol.

| | |
|---|---|
| 85% $H_3PO_4$ | 10 |
| 38% HCl | 2 |
| Water | balance |

Operating conditions: 80°±10°F.
100±50 amp./sq. ft.
2 minutes

(*b*) *Chemical pickling*
Conc. $HNO_3$ (70%)
Operating conditions: 80°±10°F.
2 minutes

(*c*) *Water rinsing*

(*d*) *Acid dipping* (for plating from alkaline baths)
$(NH_4)_2SO_4$ 100 g./L.
adjusted to pH 2 with $H_2SO_4$
balance water
Operating conditions: 80° (27°C.)±10°F.
½–1 minute

(*e*) *Water rinse*

(*f*) *Electroplating*

Aluminium, copper, nickel, iron and zinc can be satisfactorily deposited, to give adherent coatings, after completion of step (*c*) above. For electrodeposition of metals such as silver or tin, from highly alkaline solutions, steps (*d*) and (*e*) must be included in the cycle.

The direct method, although offering obvious advantages in ease of processing, has the disadvantage that preferential dissolution of stringers of inclusions is liable to occur during anodic pickling, resulting in formation of voids which are difficult or impossible to bridge.

The zinc-immersion procedure is similar to methods used in electroplating on aluminium and magnesium: the following details are given of treatments found to give good results:—

**II. Zinc-Immersion Method**

(*a*) *Zinc immersion* .. .. .. .. g./L.

| | |
|---|---|
| $Na_4P_2O_7$ .. .. .. .. .. | 120 |
| $ZnSO_4.7H_2O$ .. .. .. .. | 40 |
| NaF .. .. .. .. .. | 7·5 |
| $K_2CO_3$ .. .. .. .. .. | 5 |

pH 7·5–8·0 (adjusted with $H_2SO_4$ and $H_3PO_4$)
180°F. (83°C.)
Immersion for 5 minutes

(*b*) *Cold-water rinsing*
(*c*) *Copper plating* (from a cyanide bath)
(*d*) *Water rinsing*
(*e*) *Outgassing*, by heating for 30 minutes in boiling water
(*f*) *Cyanide dipping*: 45 g./L. NaCN
80°F. (27°C.) ±10°F.
½–1 minute
(*g*) *Water rinsing*
(*h*) *Acid dipping* in 3*N* $H_2SO_4$ at room temperature
(*i*) *Water rinsing*
(*j*) *Plating* with selected finish metal.

Details are given of baths suitable for electrodeposition of aluminium, chromium, copper, iron, nickel, silver, zinc, on beryllium surfaces suitably prepared by one or other of the procedures recommended.

For electrodeposition of nickel the recommended conditions are as shown below:—

*Strike bath* .. .. .. .. .. g./L.

| | |
|---|---|
| $NiSO_4.7H_2O$ .. .. .. .. | 143 |
| $MgSO_4.7H_2O$ .. .. .. .. | 75 |
| $NH_4Cl$ .. .. .. .. .. | 15 |
| $H_3BO_3$ .. .. .. .. .. | 15 |

with 20 mL./L of a proprietary addition agent.
Operating conditions: pH 5·5
90°F. (32°C.)
15 amp./sq. ft.

*Nickel-plating bath* g./L.

| | |
|---|---|
| $NiSO_4.7H_2O$ .. .. .. .. | 300 |
| $NiCl_2.6H_2O$ .. .. .. .. | 50 |
| $H_3BO_3$ .. .. .. .. .. | 35 |
| NaCOOH .. .. .. .. .. | 15 |

with 20 mL./L of a proprietary agent.
Operating conditions: pH 4·0
140°F. (60°C.)
40 amp./sq. ft.

## Electroplating of Zirconium

W. C. SCHICKNER, J. G. BEACH and C. L. FAUST: 'Electroplating on Zirconium.' *Jnl. Electrochemical Soc.*, 1953, vol. 100, June, pp. 289-91.

When conventional methods are used for electroplating of zirconium, the coatings produced are essentially electroformed shells, and can be easily separated or peeled from the base metal. Heat-treatment of such electroplated composites results in localized blistering or overall separation at the basis metal/coating interface. Adherent electrodeposited coatings can, however, be produced on zirconium by etching, plating with nickel or iron, pre-baking, and heating to alloy the interface layers of the two metals. Coatings of other metals may then be superimposed on the nickel or iron.

As a result of experimental work, details of which are given in this paper, the following procedure is recommended:—

1. *Descale*—
   (*a*) sandblast, (*b*) vapour blast, *or* (*c*) surface grind.
2. *Clean in alkaline solution.*
3. *Rinse.*
4. *Etch*—

| | |
|---|---|
| Solution: | $NH_4F$ 18–52 g./L. |
| | HF 3–16 g./L. |
| Molar ratio | $NH_4F$/HF 1·2–4·1 |
| Temperature | 100°F. (38°C.) |
| Time | ¾–3 min. |
| Metal removed | 0·6 mil. |
| Container | Polyethylene |

5. *Rinse.*
6. *Electroplate with Nickel*—

| | |
|---|---|
| Solution: | $NiSO_4.7H_2O$ 330 g./L. |
| | $NiCl_2.6H_2O$ 46 g./L. |
| | $H_3BO_3$ 37 g./L. |
| | $H_2O_2$ added periodically to prevent pitting |
| | pH 2·0 |
| | Temperature 140°F. (60°C.) |
| | Current density 40 amp./sq. ft. |
| | Plate thickness 1–2 mil. |

7. *Rinse and dry.*
8. *Pre-baking Treatment*—
   Temperature 400°F. (204°C.): may be carried out in air.
   Time 2–4 hours.
9. *Heat-Treatment*—
   Temperature 1300°F. (704°C.): may be carried out in air.
   Time 10–45 min.
   Quench in air or water.

Using the above method, adhesion in the as-plated condition is of the order of 6,000 p.s.i., and the thermal treatments recommended produce alloy bonding giving about 50,000 p.s.i. (as indicated by modulus of rupture).

## Nickel Plating of Aluminium Propeller Blades with Rubber Bonding

'Nickel Plating of Aluminum Propeller Blades.' *Plating*, 1953, vol. 40, July, pp. 774-6.

Aluminium and its alloys are characterized by many properties which make them suitable for aircraft propellers, but in service such as that involved in seaplanes and flying boats the constant impact of seawater spray has a steadily eroding effect on the metal. In an attempt to obviate erosion by increasing the hardness of the propeller surface nickel plating was an obvious choice, but when the nickel was deposited by conventional methods some difficulty was experienced in obtaining the quality of coating required.

The solution to the problem has been found as a result of co-operative work by the Hamilton Standard Division of United Aircraft Corporation, the Bart Laboratories, and Arthur D. Little, Inc. Bart Laboratories have developed a new hard-nickel solution which has met the requirements laid down for this, as well as for other applications, and special techniques have been evolved for production of a finished surface which more than satisfies the most stringent

specifications laid down for industrial nickel plating.

A rubber bonding material which has been treated to make it conductive is applied to the blades, and, after drying, they are plated with nickel by conventional methods, using the special hard-nickel bath. Only twenty-four hours are required for processing of a component. The finished blades have a high Vickers hardness rating, the plating is stress-free, and the surface is of a semi-bright type which can be polished chemically or mechanically to give a high lustre. This article gives a pictorial survey of the steps in preparation, rubber-coating, plating, and finishing of propellers.

Service results available to date indicate that after hundreds of landings and take-offs the condition of nickel-plated propellers demonstrates the protective character and durability of the coating, and severe tests by the U.S. Navy and by manufacturers confirm the ability of the coated blades to withstand conditions in which unplated blades were severely eroded.

Satisfactory results have been obtained with all the aluminium alloys which have been tested to date, and it is claimed that the new process opens up many applications for aluminium which were previously considered impossible or impractical, since it provides a combination of the hardness and corrosion-resistance of nickel with the advantages of aluminium and its alloys. The process is known as 'Alni-clad'.

**Building up and Reclamation of Components by Electrodeposited Nickel and Chromium**

'Some Typical Applications of the Fescol Electro-Deposition Process.' *Machinery* (*Lond.*), 1953, vol. 83, July 24, pp. 161-7.

This article describes some current applications of electrodeposited coatings produced by the Fescol process, which, since its development in the early 1920's, has been of increasing advantage to the engineering industries as a means of maintenance and repair of components of widely varying types.

Specialized methods of pre-treatment, in electrolytic etching baths, are used to ensure the high degree of adhesion which is a *sine-quâ-non* of successful use of electrodeposited coatings for such service. The procedures used have been progressively improved, and with the techniques now employed it is claimed that the deposit becomes virtually an integral part of the base metal, the two being held together by atomic, rather than by mechanical, forces. A further important characteristic of Fescol coatings is that the deposited metal is usually harder than the same material in other forms. For example, for nickel the hardness of the coating may vary between 150 and 250 V.P.N., and for chromium there is a range of 700-900 V.P.N. The combined wear- and corrosion-resistance obtained in electrodeposited coatings of this nature is used to advantage on many components, of which typical examples are described in this article. Some of the uses discussed are indicated below:

*Hydraulic rams.* Untreated rams frequently deteriorate due to contact with water, and, if the liquid is contaminated by dirt, scoring often causes excessive wear. Nickel or chromium coatings have proved most beneficial: a case is quoted of an accumulator ram, where, prior to Fescolizing, packings had to be renewed once a month, but in which, after coating with nickel, 3-5 years' service was obtained. Many makers of hydraulic equipment now make it a regular practice to have new rams treated with nickel or chromium before being put into service.

*Steel Seat and Gate for Automatic Lubrication Conduit Gate Valve.* A thickness of 0·0005 in. of chromium, applied to the faces of these components, which are in sliding contact with each other in service, and to the valve stem, has been found to prevent corrosion and pitting, which often leads to early failure of untreated valves.

*Sheet-Metal Forming and Drawing Dies.* Here the low coefficient of friction of chromium surfaces is turned to good account: it is reported that a thirty-fold increase in life, over untreated dies, is not unusual. Moulds for production of plastic components are also a successful application for chromium coating.

*Gauges.* Many thousands of gauges are treated every year, both before being put into service, and on a repair basis.

*Reclamation of Worn Components and Building up to Size.* Examples used to illustrate these important aspects of Fescolizing include a shaft nickel-coated after being worn down by fretting corrosion; a spindle, one end of which required to be enlarged; the driving gear of an aircraft engine pump; various parts of textile machines; chromium plating of broaches when the height of the teeth has been reduced by re-grinding; a heavy vehicle gear-box clutch.

Much work is done by this process in the nickel-coating of large and heavy components, such as shafts, rolls and rams up to 20 ft. long and weighing up to 6 tons. At the other end of the size scale, an extensive range of small parts is treated, e.g., new components for teleprinters, which are chromium plated to give (a) corrosion-resistance, (b) increased wear-resistance, and (c) easy movement where surfaces are in sliding contact.

In addition to deposits of nickel and chromium, lead and copper coatings are applied by the Fescol process. Reference is also made to a recent development by which chromium deposition can be carried out direct on aluminium-base components.

**Atmospheric Exposure Tests on Electrodeposited Copper-Nickel-Chromium Coatings**

AMER. SOC. TESTING MATERIALS: 'Report of Committee B 8, Sub-Committee II, on Atmospheric Exposure Tests on Copper-Nickel-Chromium Deposits on High-Carbon Steel,' contained in *A.S.T.M. Preprint 14*, June, 1953, pp. 5-15.

The 1951 report of this Sub-Committee gave inspection data on the original set of copper-nickel-chromium-plated panels which had been on exposure at various locations for approximately 3½ years, and made reference to a supplemental set of panels on which tests had then just been started.

The present report includes virtually the final inspection data on the original set of panels (after 5·2

years of exposure), and also information on the behaviour of the supplementary set, after exposure periods of 0·4, 1·4 and 2·4 years.

Full details of ratings (on the basis of corrosion, pitting, flaking, peeling, cracking, etc.) are given in tabular form, and detailed comments are made. Among the points to which attention is directed are: (1) the marked influence of the type of atmosphere in which exposure is made; (2) the influence of type of plating and thickness; (3) nickel-chromium vs. copper-nickel-chromium coatings; (4) the effect of heavy rainfall on rate and degree of deterioration of the coatings. In connexion with (4) it has been decided to incorporate, in some exposure tests now being planned, a regular washing cycle on portions of exposed panels, to simulate more nearly natural atmospheric-exposure conditions.

### Influence of Chromium Plating on Fatigue Limit of Nickel-Chromium-Molybdenum Steel

G. M. CABBLE: 'The Effect of Chromium Plating of Steel on the Fatigue Limit.' *Metal Finishing*, 1953, vol. 51, June, pp. 106-8; July, pp. 60-3.

Locomotive side rods of A.I.S.I. 4340 steel which had been chromium-plated at the lateral faces of the bearing opening (to obtain increased wear-resistance) showed, while in service on the Norfolk and Western Railway, U.S.A., failure of a type which suggested that the fatigue limit of the steel had been lowered by chromium plating. The Engineering Experiment Station of the Virginia Polytechnic Institute was commissioned to investigate the matter, and this article is based on the report issued in their Bulletin No. 82.

The practice had been to apply chromium plating about 0·010-0·015 in. thick, directly on to the basis metal, all other portions of the rod being blocked off. After the rods had been in service and the plating had been worn down by normal wear, the faces were replated without removing the old plating.

A review of the pertinent literature revealed that chromium plating reduced the fatigue limits of all types of steel which had been tested after such treatment, but that no work had been done on the effect of more than one plating. It was therefore decided to carry out an investigation on the 4340 steel to determine the effect, on fatigue limit, of—

(1) a single plating with chromium;

(2) a baking cycle after chromium plating;

(3) a second plating, after the first had worn down;

(4) a baking treatment, after the second plating.

Full details are given of the experimental conditions used, and the results are fully reported and discussed.

| *Condition* | *Fatigue Limit* | |
|---|---|---|
| | p.s.i. | t.s.i. |
| Unplated .. .. .. .. | 73,000 | 32·55 |
| Chromium-plated once .. .. | 57,500 | 25·65 |
| " " " , baked once | 72,500 | 32·35 |
| " " twice .. .. | 50,500 | 22·55 |
| " " " , baked twice | 69,500 | 31·05 |

The values of the fatigue limits for the 4340 steel in the unplated condition and after the respective treatments are shown in the Table.

It is observed that for the once-plated, unbaked, condition, for which earlier data are available, the author's results agree well with those reported by other investigators.

### Tin-Nickel Alloy Plating: Current Stage of Development

J. W. CUTHBERTSON: 'The Case for Tin-Nickel Alloy Plating.' *Metal Industry*, 1953, vol. 83, July 31, pp. 89-91.

Some three years ago the Tin Research Institute introduced a new type of electrodeposited coating, consisting of an alloy containing about 65 per cent. of tin with 35 per cent. of nickel. Although several technical papers relating to the process have been published, no detailed account has been given of exploitation of the process on an industrial scale. The present article discusses some difficulties which have arisen in large-scale operation, and methods which have been adopted to overcome them. Aspects dealt with include the constitution of the alloy coating, filtration procedure for prevention of pitting, fume extraction, behaviour of the nickel and the tin anodes and general operational control during deposition. The present position of development is reviewed, with emphasis on the advantages of the process.

### Nickel-Dip Process

The following brief abstracts have been published of papers presented at the April 1953 meeting of the American Ceramic Society:—

D. G. MOORE, J. W. PITTS and W. N. HARRISON: 'An Investigation of the Effects of the Nickel Dip on the Adherence of Ground Coat Enamels to Steel.'
*Bull. Amer. Ceramic Soc.*, 1953, vol. 32. Apr., p. 150.
'The effects of firing temperature and time on the adherence index of nickel-dipped enamelling iron and titanium-bearing steel were determined for a cobalt-free and a cobalt-bearing ground coat. In addition, such variables as weight of nickel deposit, and surface pre-treatment of the steel, were investigated. Numerous experiments are described.'

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J. C. RICHMOND, H. B. KIRKPATRICK and W. N. HARRISON: 'A Radioisotope Study of the Nickel Dip,' *ibid.*, p. 150.

'Specimens were treated in a nickel-dip solution containing radioactive nickel 63 in the form of nickel ammonium sulphate. Uncoated specimens were examined by autoradiography and by counting techniques, to study the distribution of the radioactive nickel. Other specimens were enamelled and examined by autoradiography as well as by chemical and counting techniques, to determine the distribution of the radioactive nickel in the coated specimens. Evidence of non-uniformity of nickel deposit is presented.'

(The work reported in both papers was carried out at the National Bureau of Standards, Washington, D.C.)

**Electroless Plating: the Kanigen Process**

'Electroless Plating.' *Production Engineering*, 1953, vol. 24, May, pp. 221-3.

See also—

J. B. CAMPBELL: 'Electroless Nickel Plating: Where it Stands To-day.' *Materials and Methods*, 1953, vol. 37, May, pp. 96-9.

These articles contain a summary of facts, technical and commercial, with regard to a chemical coating process (Kanigen process) which is being used for deposition of hard wear-resisting coatings on industrial components such as valve bodies, camshafts, and crankshafts, tank linings, piping, cams, bearings, and pinions. Some of the main items of information recorded are given below:—

The basic research from which the process was developed was carried out at the National Bureau of Standards; the essential features are covered by two Government-owned patents, U.S. 2,532,283 and 2,532,284. The process has been commercially developed by General American Transportation Corporation. It consists essentially of immersion of the part which is to be coated in an agitated solution of nickel chloride and sodium hypophosphite, held at 100°C. The deposition process depends on catalytic reduction of nickel compounds by the hypophosphite, on the surface of certain metals: the coating deposited is a nickel-phosphorus alloy. As deposited it has normally a hardness of 550-650 Vickers: by variations in plating technique and/or heat-treatment, hardnesses as low as 400 or as high as 900 Vickers can be produced.

The baths developed by Brenner and Riddell are basically those which are in use to-day:—

| *Alkaline Bath* | oz./gal. |
|---|---|
| Nickel chloride ($NiCl_2.6H_2O$) .. .. .. | 4 |
| Sodium hypophosphite .. .. .. .. | 1·3 |
| Ammonium chloride .. .. .. .. | 6·7 |
| Sodium citrate .. .. .. .. .. | 13·4 |
| *Acid Bath* | |
| Nickel chloride .. .. .. .. .. | 4 |
| Sodium hypophosphite .. .. .. .. | 1·3 |
| Sodium hydroxyacetate .. .. .. .. | 6·7 |
| Brighteners (optional) | |

It is, however, noted that the bath composition used in the Kanigen process, as operated by General American Transportation Corporation, has not been disclosed. It is believed that important changes may have been made in composition of the solution, including the nature of the buffering agent.

Deposition of nickel by this process is most successful on nickel, cobalt, gold, aluminium, steel, and palladium, all of which catalyze the essential reaction. Some other metals, e.g., copper, brass, silver, must be prepared, (1) by touching, while immersed in the plating bath, with one of the catalytic metals, or (2) by pre-treatment in a dilute solution of a palladium salt. Lead and cadmium poison the reaction, and compounds of these metals in the bath will prevent deposition even on catalytic metals. In plating of lead, cadmium, or zinc by the electroless process an undercoat of copper or other catalytic metal must be deposited. It is claimed that glass and plastics can be successfully treated, giving satisfactory adhesion. Although it is expected that the process may be extended to coating of certain other metals, it is considered doubtful whether it could ever be adapted for the plating of active metals such as chromium.

The nickel-phosphorus coating is highly resistant to wear and to corrosion: its appearance depends largely on the finish of the surface on which it is deposited.

It is noted that additional user patents have been granted, in the U.S.A., e.g., to (1) Ferro Corporation, for use of the electroless process as a pre-treatment of steel, to give improved adhesion of vitreous enamel coatings, and (2) Wright Aeronautical Division of Curtiss-Wright Corporation, for plating of piston ring grooves on aluminium pistons. The method is also in use for plating of the interior of pumps. The possibility of coating parts of unusual size and shape indicates the likely extension of the Kanigen process to plating of many engineering parts, and General American Transportation Corporation is currently extending its plant with a view to undertaking plating work for various industries. It is probable that at a later stage the process will be licensed by them, with inclusion of rights under improvement and equipment patents for which application has already been made.

**Stripping of Nickel from Plated Parts**

C. D LEONARD: 'The Stripping of Plating Shop Rejects: A Summary of Recommended Methods.' *Electroplating*, 1953, vol. 6, Aug., pp. 290-2, 296.

The survey opens with a note on faults liable to necessitate rejection of electroplated parts, and with general recommendations on choice and operation of stripping processes. Specific recommendations are then made with regard to electrolytic or chemical-immersion procedure for stripping of nickel, chromium, copper, cadmium, zinc, silver, gold, tin and tin alloys, lead, platinum-group metals. The following methods are considered for nickel:—

1. *Sulphuric Acid.* The most common method for stripping nickel deposits from iron, steel, copper and copper alloys, zinc, and aluminium alloys is by making the work anodic in 60-80 per cent. (by vol.) sulphuric acid to which 0·5-1·5 per cent. of glycerin is added as inhibitor. The tank should be earthenware or lead, and lead cathodes are used. Current density 50-100 amp./sq. ft. at 6-12 v. Minimum attack on the basis metal is secured by operation at room temperature, and to counteract the heating effect of the current, cooling by water coils may be required if a large volume of work is being handled. When stripping nickel from zinc and aluminium alloys it is preferable to use concentrated sulphuric acid, and to make contact before the work is immersed.

2. *Hydrochloric acid.* Nickel may be stripped from copper and brass by anodic treatment in hydrochloric acid (10 per cent. by vol.) at 6-12 v., at room temperature. Gas-carbon cathodes are used, with earthenware

or rubber-lined tanks. The rate of stripping by this procedure is considerably faster than in sulphuric acid, but there is usually more attack on the basis metal.

3. *Nitric acid.* Nitric-acid baths have been used for stripping nickel from steel and aluminium because both the latter metals are passive in the concentrated acid. If water is introduced into the solution, steel surfaces will be pitted.

4. *Sodium nitrate.* Nickel may be stripped from iron and steel by treatment in 50 per cent. sodium-nitrate solution in water, used at 100-200 amp./sq. ft. and 200°F. (93°C.), with steel cathodes. High current density and operating temperature are necessary, but the method gives good results.

5. *Nitric-sulphuric acid.* The normal bright-dipping solution may be used for stripping nickel from some materials, e.g., brass or nickel silver. The solution is, however, liable to attack the basis material.

**Sprayed Stainless-Steel Coatings**

See abstract on p. 159.

## NON-FERROUS ALLOYS

**Alternating-Current Resistance Standards: Use of Nickel Alloys**

A. H. M. ARNOLD: 'Alternating-Current Resistance Standards.' *Proc. Instn. Electrical Engineers*, 1953, Part II, vol. 100, June, pp. 319-28.

This paper deals in detail with the basic design principles covering resistance standards which have a resistance differing from the nominal value by less than 0·01 per cent. and a phase angle of less than 0·0001 radn. at frequencies up to about 20 kc/s.

The first section of the paper includes particulars of the properties of alloys which have been used for resistors of the type considered. These are:—

Copper-nickel (60-40 per cent.): Constantan type.

Copper-manganese-nickel (84-12-4 per cent.): Manganin type.

Nickel-chromium-aluminium (75-20-2 per cent.), produced under the names of Evanohm and Karma, the former containing also about 2 per cent. of copper, the latter about 3 per cent. of iron.

Reference is also made to exceptionally low values of temperature coefficient which can be developed in gold-chromium alloys, but the cost of such alloys, and the fact that commercial availability has not yet been established, preclude their consideration in the present connexion.

It is urged that the nickel-chromium-aluminium alloys, on account of their small change of resistance with temperature, appear to be particularly suitable for standards which must be operated with a considerable rise of temperature due to self-heating.

The author gives formulae for calculation of the residual inductance of standards of various types. He describes a one-ohm standard having a calculated phase angle of less than 0·0001 radn. at frequencies up to 30 kc/s.

**Nickel-base Oxide Cathodes**

See abstract on p. 129.

**Temperature-Sensitive Magnetic Nickel Alloys**

See abstract on p. 139.

**Wrought Corrosion-Resisting Nickel-Molybdenum Alloys**

See abstract on p. 156.

**Spontaneous Magnetization in Nickel-Copper Alloys**

D. J. OLIVER and W. SUCKSMITH: 'The Spontaneous Magnetization of Alloys. I. Copper-Nickel Alloys.' *Proc. Royal Soc., Ser. A*, 1953, vol. 219, Aug. 11, pp. 1-17.

Spontaneous magnetization is of fundamental importance in relation to the theory of ferromagnetism, but only meagre experimental data on the property are at present available. This paper describes the initial stage of a project planned to determine the temperature variation of spontaneous magnetization for a number of ferromagnetic materials. An alloy of copper and nickel was chosen (containing 23·9 at. per cent. copper, remainder nickel) because this system occupies an important position in the electron theory of ferromagnetism, and has the additional advantage of freedom from metallurgical complications, since it forms a series of solid solutions over the entire range.

The work is described in two parts, concerned, respectively, with comparison of methods used for deducing spontaneous magnetization from experimental observations, and to the spontaneous magnetization/temperature characteristics of the individual alloys studied.

**Thermal Conductivity of Nickel and Nickel Alloys: Summary**

See abstract on p. 128.

**Sea-Water Tests on Beryllium-Copper, Cupro-Nickel and Other Alloys**

See abstract on p. 159.

**Nickel Alloy Coinage**

See abstract on p. 126.

**Precipitation-Hardening Copper-Nickel Alloy for Springs**

'Substitute for Copper-Beryllium.' *Metal Industry*, 1953, vol. 83, July 31, pp. 87-8.

This note gives details of a range of copper-nickel alloys containing silicon and aluminium, developed at Battelle Memorial Institute for International Business Machines Corporation. Brief reference was made to these materials in *Steel* of March 30, 1953:

see abstract in *Nickel Bulletin*, 1953, vol. 26, No. 6, pp. 94-5. Additional particulars in *Metal Industry* include compositions of alloys studied in developing the optimum composition of nickel 10, aluminium 4, silicon 1·5, per cent., balance copper.

**Colour and Reflecting Properties of Stainless Steels and Other Materials**

See abstract on p. 153.

**Relation between Magnetic Properties and Structure in Nickel-Manganese Alloys**

G. R. PIERCY and E. R. MORGAN: 'Ferromagnetism and Order in Nickel-Manganese Alloys.'
*Canad. Jnl. Physics*, 1953, vol. 31, May, pp. 529-36.

Twenty alloys containing amounts varying up to 40 at.-per cent. of manganese were subjected to heat-treatments designed to produce an atomic arrangement varying from disorder to a high degree of long-range order.

The degree of order of $Ni_3Mn$, calculated from measured saturation magnetization, using the atomic model of ferromagnetism, was consistent with the value calculated from the ratio of measured integrated intensity of the (110) X-ray diffraction superlattice line to that of line (111).

The relation between saturation magnetization and concentration for the disordered alloys can be explained adequately by the existence of short-range order. A value of 3·4 Bohr magnetons for the effective magnetic moment of manganese atoms in a nickel lattice was deduced.

**Electrical Resistivity of Cobalt-Nickel Alloys**

T. BROOM and C. S. BARRETT: 'The Effect of Deformation on the Electrical Resistivity of Some Cobalt-Nickel Alloys.' *Acta Metallurgica*, 1953, vol. 1, May, pp. 305-9.

Resistivity determinations on annealed and on deformed wires of cobalt-nickel alloys (nickel 25·9, 31·1 and 41·1 per cent., respectively) have confirmed conclusions drawn from earlier investigations, that stacking faults can make a large direct or indirect contribution to the increase in resistivity resulting from cold work. Although the relative densities of stacking faults in the various alloys had generally to be deduced from known transformation characteristics, X-ray evidence was obtained by the authors for the presence of stacking faults in the deformed alloy which showed the largest increase in resistivity.

**Hall Effect in Cobalt-Nickel Alloys and in Nickel**

S. FONER and E. M. PUGH: 'Hall Effects of the Cobalt-Nickel Alloys and of Armco Iron.' *Physical Rev.*, 1953, vol. 91, July 1, pp. 20-7.

Experimental investigations of the Hall effects in cobalt, cobalt-nickel alloys and Armco iron are described in detail. Precision measurements were made with magnetic fields up to 30 kilogauss, in order to separate the ordinary from the extraordinary effects. The ordinary effect in cobalt-nickel alloys at intervals throughout the system was found to be negative, and agreed within a factor of two with values expected from only 4*s* band conductivity. Some new data on nickel are also presented.

**Colorimetric Determination of Iron in Nickel-containing and Other Materials**

J. A. DEAN and J. H. LADY: 'Colorimetric Determination of Iron with Nitroso R Salt.'
*Analytical Chemistry*, 1953, vol. 25, June, pp. 947-9.

Although it was observed as early as 1921 that nitroso R salt gave a bright green colour reaction with very dilute solutions of ferrous iron, little has since been done towards applying this reaction to the colorimetric determination of iron, due to the fact that many of the other elements usually present in alloys of various types also form highly coloured complexes with the salt.

The present paper describes means for the elimination of interference by other elements. Hydrazine is used as a reducing agent, and the temperature is maintained at 75°-80°C., thus ensuring destruction of all diverse ion complexes and any excess dye. Full details are given of the procedure optimum for determination of iron in various types of alloy, including nickel silver, Monel, nickel-chromium alloys, and various copper- and aluminium-base alloys.

**Powder-Metallurgy Symposium**

See abstract on p. 126.

## NICKEL-IRON ALLOYS

**Temperature-Sensitive Magnetic Nickel Alloys**

T. A. HEDDLE: 'On the Temperature Sensitivity of Special Magnetic Materials.' *Brit. Jnl. Applied Physics*, 1953, vol. 4, June, pp. 161-6.

This paper gives a survey of the properties and uses of magnetic materials which are sensitive to temperature in the range $-60°$ to $+120°$C.

Two main types of thermomagnetic material are in commercial production:—alloys having the approximate compositions 70-30 per cent. iron-nickel and 70-30 per cent. nickel-copper. The Curie points and intensities of saturation magnetization are graphically illustrated for the two groups. The thermomagnetic characteristics of homogeneous and of heterogeneous alloys are discussed in relation to their utilization for different degrees of control, and problems arising through irreversibility of certain alloys are considered.

Reference is made to commercially available alloys representative of the iron-nickel and nickel-copper alloys which are of interest in thermomagnetic control:

*Iron-Nickel:* Mutemp, R.2799, N.I.F.E. 30, Compensator Alloy, Temperature Compensator Alloy N.30, Thermoperm, N.M.H.G., Varioperm, and Shunt Steel.

*Nickel-Copper*: JAE Metal, Calmaloy, Westinghouse Alloy.

(In this section reference is also made to certain

other alloys which have Curie points at temperatures falling within the range under consideration, but in these cases the commercial forms of the alloy are not controlled so closely as to permit practical utilization of this property).

Sources of supply of the proprietary alloys are given.

Applications of the thermomagnetic materials are discussed in two groups: (1) those used in electromagnetic devices which are intended to respond to temperature variations, and (2) those employed in electromagnetic instruments or apparatus requiring compensation for temperature errors. Typical examples are described.

### Studies of Ideal Magnetization in Nickel and Nickel Alloys

L. A. CHENIKOVA: 'The Investigation of the Ideal Magnetization Curve for Soft Polycrystalline Ferromagnetic Materials.' *Zhur. Eksper. Teoret. Fiziki*, 1951, vol. 21, pp. 514-23.

Measurements were made on specimens of molybdenum-Permalloy (nickel 78, molybdenum 3·8, iron 18, per cent.), copper-molybdenum-Permalloy (nickel 74, copper 8·8, molybdenum 3, per cent., balance iron), Armco iron, and nickel, using ballistic methods on ring specimens and the magnetometer method on wire. Details of methods used, and of results obtained, are summarized in *Metallurgical Abstracts*, 1953, vol. 20, June, p. 683.

### High-Permeability Nickel-Iron Alloys containing Copper and Molybdenum

F. ASSMUS and F. PFEIFER: 'Alloys of Very High Permeability in the Nickel-Iron-Copper-Molybdenum Series.' *Metall*, 1953, vol. 7, Mar., pp. 189-91.

BOOTHBY and BOZORTH have described a treatment by which specially high initial permeability can be produced in a particular alloy in the Permalloy series (nickel 79, molybdenum 5, manganese 0·5, per cent.): see *Jnl. Applied Physics*, 1947, vol. 18, p. 173. The alloy has been given the name of 'Supermalloy'.

An essential feature of this treatment is a low-temperature tempering, at 400°-500°C., or cooling at a controlled rate, but it is also imperative that raw materials of very high purity be used, that certain conditions be observed in melting, and that the alloy be annealed in pure hydrogen prior to the low-temperature treatment.

The authors of the present paper report experiments carried out to determine whether specialized heat-treatment of a similar type could confer, on other alloys of nickel-molybdenum-iron and of nickel-iron-molybdenum-copper types, similar properties of outstandingly high initial permeability.

Examination of a series of alloys containing nickel 68-81 per cent. approx., with iron and various amounts of molybdenum, or copper + molybdenum, subjected to the cycle of treatment found optimum for Supermalloy, indicates that development of specially high initial permeability is possible in a relatively wide range of compositions. Particularly high values were obtained, after the controlled heat-treatment, in alloys of the following compositions:—

| Nickel % | Copper % | Molybdenum % | Iron % | Initial Permeability |
|---|---|---|---|---|
| 79·25 | — | 4·8 | bal. | 91,000 |
| 78·05 | 2·0 | 5·1 | bal. | 90,000 |
| 76·3 | 5·0 | 3·15 | bal. | 92,000 |
| 76·55 | 5·0 | 5·5 | bal. | 110,000 |
| 73·65 | 10·2 | 3·2 | bal. | 105,000 |
| 71·0 | 14·0 | 3·0 | bal. | 92,000 |

### Nickel and Nickel Alloys in Electronic Equipment

See abstract on p. 128.

### Magnetic Nickel-Iron Alloys in Passenger Lift Controls

'Passenger Elevator Controls Use 50% Nickel Alloy.' *Nickel Topics*, 1953, vol. 6, No. 6, p. 5.

This short article describes the use of Hipernik (50-50 nickel-iron alloy) in the Rototrol system incorporated in Westinghouse lifts, for control of acceleration, deceleration and stopping position. The advantages of the system are described: these include minimum loss of time in stopping and starting, smooth stopping, and good levelling to the respective floors. Utilization of the magnetic properties of the nickel-iron alloy has made a significant contribution to all these important features.

### Nickel Alloys in Glass-to-Metal Seals

HENRY WIGGIN AND CO., LTD.: 'Glass-to-Metal Seals.' *Publn.* 307*b*; 10 pp.

Types of seal used in various forms of electrical and other equipment are described, with illustrations. The characteristics of the glasses employed in such apparatus are reviewed, and an account is given of alloys which have been developed to give the thermal-expansion values and other properties matching them to the respective types of glass.

Alloys employed for such purposes include a range of nickel-iron materials in which the proportions of the constituent basis elements and of special additions are closely regulated to ensure accurately controllable properties compatible with those of glasses of soft or hard grades. The mechanism of sealing is described, and precautions required to secure satisfactory operation of the glass-metal composites are reviewed. Stresses set up in various types of seal are briefly discussed.

The revised form of the publication (superseding 307 and 307a) has been brought up to date in the light of new information on thermal expansion which has only recently become available, and additional data on alloys of the Nilo controlled-expansion series are included.

### Manufacture of Nickel Alloy Coil Springs for Watches

C. HAAS: 'Manufacture of Coil Springs for Watches.' *Wire Industry*, 1953, vol. 20, June, p. 601; from *Die Uhr*, 1952, vol. 6, pp. 26-8, 54.

The author describes the specialized production technique used for making coil springs of watches from the corrosion-resisting nickel-base alloy Nivarox. Particulars are given of wire drawing, rolling, and winding operations, and attention is directed to the importance of final heat-treatment of the formed springs, as a factor influencing their quality and service characteristics.

### Measurement of Thermal Expansion of Invar Geodetic Surveying Tapes

P. HIDNERT and R. K. KIRBY: 'A New Method for Determining Linear Thermal Expansion of Invar Geodetic Surveying Tapes.' *Jnl. of Research, Nat. Bur. Standards*, 1953, vol. 50, Apr., pp. 179-85; R.P. 2407.

Determinations of the linear thermal expansion of Invar geodetic tapes have been regularly carried out at the National Bureau of Standards for some 50 years. Up to the present time measurements of the lengths of the tapes have been made at several different constant temperatures, ranging from about 5° to 35°C. The constant temperatures were obtained by changing the ambient temperature of the entire laboratory in which the determinations were made. From these measurements of length and temperature coefficients of linear thermal expansion were derived.

The work described in this paper was undertaken to develop a new method for determining coefficients of linear thermal expansion without the necessity of changing the ambient temperature of the whole laboratory. In the new procedure the tape is heated by passing through it direct current, while the ambient temperature is held constant. Observations of changes in length and electrical resistance are taken at various constant temperatures of the tape. The temperature coefficient of resistance of each tape is obtained experimentally or from a relationship, derived by the authors, between temperature coefficients of resistance and mass resistivities. The actual temperatures of the tape are determined from its different resistances and temperature coefficients of resistance. Coefficients of expansion determined by this method, from 15° to 35°C. are in good agreement with those obtained by the method previously used at the Bureau. With the new method greater precision is obtained in a much shorter time.

### Thermal Conductivity of Nickel and Nickel Alloys: Summary

See abstract on p. 128.

### Powder-Metallurgy Symposium

See abstract on p. 126.

### Determination of Copper in High-Alloy Steels and Cast Irons

See abstract on p. 157.

## CAST IRON

### Electrical Resistivity of Cast Iron: Influence of Composition, etc.

K. B. PALMER: 'The Electrical Resistivity of Cast Iron.' *Brit. Cast Iron Research Assocn., Jnl. of Research and Development*, 1953, vol. 4, June, pp. 571-85; B.C.I.R.A. Research Report 359.

Part I of this report contains a summary of the literature on electrical resistivity of cast iron, supported by bibliographical references: Part II describes results of determinations made on bars of various compositions, cast in the Association's laboratories. In each case the property of electrical resistivity is considered in relation to composition, and an evaluation is made of the influence of the respective normal and alloy elements present in cast iron:

The conclusions, drawn from the information available from the literature and from the experimental work now reported, are as follows:—

'Carbon and silicon have by far the greatest influence on the electrical resistivity of cast iron. The lower the carbon and silicon contents, the lower will be the resistivity of the iron, and as the carbon and silicon are increased the resistivity increases.

'When carbon is present as graphite, coarse flake structures give the highest resistivity, with a lowering of resistivity as the flakes become finer: nodular graphite structures give the lowest resistivity of all.

'Manganese and sulphur do not affect the resistivity of ordinary grey cast irons, which usually have the sulphur balanced by manganese.

'With phosphorus contents up to 0·2 per cent., the effect on electrical resistivity is almost negligible, but above 0·2 per cent. the phosphorus has an effect on the graphite structure, which produces an increase in resistivity.

'Of the alloying elements, nickel and aluminium are the only ones which have any appreciable effect, both raising the resistivity with increasing additions of the element. The other elements, copper, molybdenum, chromium and vanadium, have relatively little effect on the electrical resistivity beyond a slight tendency to lower it.

'The principal factor, however, with respect to composition is the resulting structure of the iron due to the presence of these elements, since the structure plays an important part in electrical resistivity. In this respect, pouring temperature and rate of cooling must also be taken into consideration.'

### Nickel-containing Cast Iron for Ball-and-Socket Joints Operating at High Temperature

'New Alloys: High-Temperature Alloy ('Rynalloy') Developed to Meet Unusual Needs.' *Iron Age*, 1953, vol. 171, June 18, pp. 172, 176, 178.

Notes on 'Rynalloy', a nickel-chromium-silicon cast iron developed by Ryan Aeronautical Company for ball-and-socket joints operating at temperatures up

to 1800°F. (980°C.) in aircraft exhaust systems. Operation of these parts is described and illustrated, and factors determining the composition of the alloy chosen for such service are discussed.

Ball-and-socket joints consist of three parts: (1) the 'ball' is a spherical section having a cylindrical hole through it, which is retained in (2) a stainless-steel tube flared at one end into (3) a close-fitting cup. Within the ball another tube is inserted and flared to form a close-fitting assembly which permits the connecting tubes to slip, twist, and rotate with universal action. As many as 56 ball-and-socket joints are used to isolate the vibration of large power plants employed in modern aircraft.

Components of the ball-and-socket joints must fit together closely, with a large area of metal-to-metal contact. In service the metals rub together constantly, due to vibration and thermal movement, and at the high temperature involved no lubricant can be provided. Under such conditions one of the first requirements for the ball material is good resistance to oxidation: other limitations are imposed by the need for a coefficient of thermal expansion matching that of the stainless steel with which the ball is in contact. The material selected as having the desired properties is a high-alloy cast iron containing nickel about 20, chromium not less than 1·8, silicon up to 6, carbon not more than 2·5, manganese not more than 1, per cent., with sulphur as low as possible. Graphitic carbon, deposited along the grain boundaries of the materials, is necessary to provide anti-galling properties. A year of service of Rynalloy in the ball-and-socket joints has confirmed its suitability for this type of service.

### Ni-Hard Grinding Balls

K. A. DELONGE: 'Alloy Iron Balls Set New Standard in Cement Grinding.' Reprint from *Pit and Quarry*, Feb., 1953; 2 pp.
Issued by International Nickel Co., Inc.

As a result of successful use of Ni-Hard (nickel-chromium white cast iron) in liners, pumps and other portions of plant employed in the cement industry, research was undertaken some years ago on production of grinding balls.

Work was at first concentrated on balls of small size ($\frac{5}{8}$-in. to 1-in. diameter). Balls within this range could best be produced by casting in sand moulds, and procedure ensuring sound balls, free from internal shrinkage, was soon developed. Experimental work demonstrated, at this stage, that the full alloy content of Ni-Hard as normally produced (nickel 4·5, chromium 2, per cent.) was not required for the balls; a composition of nickel 3·0, chromium 1·25, per cent. proved to be optimum, consistently producing the martensitic microstructure which is essential for abrasion-resistance.

Balls to this specification were produced in quantities for test purposes and were distributed to a number of cement companies to be tried out in commercial-service conditions, in comparison with forged-steel and unalloyed-iron balls, working under parallel conditions. Some typical reports received from various sources are summarized below:—

In grinding clinker, over a period of one year, consumption of the Ni-Hard balls was only one-fourth to one-fifth of that of forged-steel balls. Tests at six other plants, extending over one- to three-year periods, confirmed this record.

Testing of alloyed cast iron balls in the grinding of wet cement stone is not yet sufficiently far advanced to permit of final conclusions, but indications to date are that the Ni-Hard balls will show a superiority over hardened steel, comparable with the performance they have already given in wet grinding of various types of metallic ore.

Some calculations are made by the author of this note as to the economies to be effected by use of the alloy cast iron balls.

### Ni-Hard for Handling Hot Clinker

'Ni-Hard Resists Hot Clinker Abrasion in Paint Plant.' *Nickel Topics*, 1953, vol. 6, No. 6, p. 6.

This note reports experience in the Pigment Division of the Imperial Paper and Color Company, Glen Falls, N.Y., which is the largest chemical pigment colour-producing unit in the world. In one of the manufacturing operations sodium dichromate is produced by roasting chromite ore at 2200°F. (1205°C.), in two rotary oil-fired kilns. The resulting hot clinker is discharged continuously onto a baffle plate, which directs it onto a conveyor at a rate of about 6 tons per hour. Ni-Hard plates 2 ft. × 2½ ft. × 1 in. thick are used to line the bottom of the conveyor trough along which the hot chromate clinker is dragged by steel scraper bars mounted on link-belt side chains. The Ni-Hard plates are exposed to the hot clinker over about one-half of the 60-ft. length of the conveyor, and it is in this zone, as well as on the incline to the discharge point, that most severe abrasion is encountered. Ordinary steel plate, formerly used, failed after relatively short service, under the combined action of heat and abrasion, but Ni-Hard plates installed in July 1951 still show few signs of wear and are expected to give many years of useful service under these severe conditions.

### Relationship of Composition to Mechanical Properties in S.G. Iron

C. C. REYNOLDS, C. M. ADAMS and H. E. TAYLOR: 'Prediction of Mechanical Properties from Chemical Composition for Fully Annealed Ductile Cast Iron.' *Amer. Foundrymen's Soc., Preprint 53-51*, May, 1953; 6 pp.

Studies of the influence of changes in chemical composition, on mechanical properties of fully ferritic S.G. iron, have led the authors to the conclusion that a precise relationship exists, and that properties can be calculated from the composition of correctly made annealed S.G. iron. The relationships are valid only for irons free from inclusions. The data are summarized in a table showing change in tensile and yield strengths, hardness, reduction of area, and elongation, per 1 per cent. increase in carbon, silicon, nickel,

manganese, phosphorus. The influence of each element was found to be independent and consistent: equations for calculation of properties from composition are developed.

**Properties and Applications of S.G. Iron**

'Spheroidal Graphite Cast Iron—Engineering Properties and Applications.'
*Mond Nickel Co., Ltd., Publn.* 630*.

This is a revised edition of Publication 603, issued in 1952 (see *Nickel Bulletin*, 1952, vol. 25, Nos. 8-9, p. 208). The text has been modified and amplified in the light of information which has become available since publication of the first edition, and some rearrangement has been done, to make the reference data more easily accessible.

A useful addition has also been made, in the form of a list of typical applications of S.G. iron. This list is classified according to the main engineering industries in which S.G. iron castings are used, and the individual applications are correlated with information given in the text on the properties which render this material specially suitable for the respective fields of service.

## CONSTRUCTIONAL STEELS

**Continuous-Cooling Transformation Diagrams for Nickel-Alloy Steels**

W. STEVEN and G. MAYER: 'Continuous-Cooling Transformation Diagrams of Steels.'
Reprint from *Jnl. Iron and Steel Inst.*, 1953, vol. 174, May, pp. 33-45; see abstract in *Nickel Bulletin*, 1953, vol. 26, No. 7, p. 115.
*Mond Nickel Co., Ltd., Publn.* 668*; 1953; 13 pp.

**Influence of Nickel and Molybdenum on Austenite-Martensite Transformation**

D. J. BLICKWEDE: 'Effect of Nickel and Molybdenum on Stabilization of the Austenite-Martensite Transformation.' *Jnl. of Metals*, 1953, vol. 5, July: *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 922-3.

The author reports work on the influence of the two elements named on stabilization in hyper- and hypo-eutectoid steels. The two series of nickel-containing steels had, respectively, carbon contents of 1·2 and 0·4 per cent., and the nickel was varied in both cases between 0 and 6 per cent. Stabilization in the forged, hot-rolled, annealed and centreless-ground samples was measured by ageing brine-quenched specimens (austenitizing temperature 1800°F.: 980°C.) at 95° and 150°F. (35° and 65° C.), and subsequently determining the change in specific volume produced by cooling from room temperature to −320°F. (−196°C.).

In 0·4 per cent. carbon steels nickel up to 3 per cent. has little, if any, effect on rate or amount of austenite stabilized at 95°F. (35°C.). Increasing the nickel from 3 to 6 per cent. doubles the amount of austenite stabilized in a given period. In 1·2 per cent. carbon steels aged at 95°F. (35°C.) increase of nickel from 1 to 3 per cent. appears to decrease the amount of stabilization, and a further increase to 6 per cent. completely eliminates stabilization. The same effects were observed at corresponding nickel levels in steels stabilized at 150°F. (65°C.), especially after ageing for periods longer than 5 hours. It thus appears that nickel up to 1 per cent. has little effect on stabilization either at low or at high carbon levels, whereas more than 1 per cent. increases stabilization at low carbon contents and decreases it with high carbon. The author discusses the explanation of these diverging effects in high- and low-carbon steel on the basis of the reaction-path theory of the austenite-martensite transformation.

In the molybdenum series 1·2 per cent. carbon steels only were used: the molybdenum contents of the two steels studied were, respectively, 0·29 and 1·00 per cent. In contrast to nickel, molybdenum markedly increases stabilization at this carbon level, an observation which is in accord with published results relating to the influence of chromium, and with theoretical considerations.

**Thermal Stabilization of Austenite in Nickel Steels**

T. KO and B. EDMONDSON: 'Thermal Stabilization of Austenite in Nickel Steels.' *Acta Metallurgica*, 1953, vol. 1, July, pp. 466-7.

It is known that in certain steels the amount of martensite obtained on quenching specimens to a fixed reference temperature is dependent on the rate of cooling from the austenitizing temperature. Morgan and Ko showed that in a steel containing carbon 1·1, nickel 5·4, per cent. the difference in the martensite contents of specimens quenched in brine and in oil was due to thermal stabilization occurring in the region 300°-400°C. Stabilization of the austenite occurred on isothermal holding at temperatures lower than this, both above and below *Ms*, but the change was too slow at these temperatures to reach a detectable amount even during oil quenching (*ibid.*, 1953, vol. 1, pp. 36-48: *Nickel Bulletin*, 1953, vol. 26, No. 4, p. 56).

The present note reports results obtained in repetitive experiments. They showed that:—

(1) The amounts of martensite in the specimens were independent of the quenching medium (brine, mercury, or oil), i.e., the cooling rate had no effect on the progress of transformation.

(2) Ageing above *Ms* (87°C.), at 178° or 214°C., for various periods, had no effect on the amounts of martensite at temperatures between *Ms* and 15°C.

(3) Stabilization was observed on ageing below *Ms*. Holding for 24 hours at 52°C. (31 per cent. martensite) caused a temperature lag of 16°C. and a loss of 15 per cent. martensite when the transformation re-started.

These results are consistent with the previous finding that any difference in the amount of retained austenite in this type of steel is associated with stabilization above *Ms*, but it is obvious that some factors were in

* *We shall be pleased to supply a free copy of this publication.*

operation preventing occurrence of stabilization above *Ms* in the specimens used. Consideration of the authors' results and of anomalies recorded by some other investigators indicate that there are some factors at present unknown which determine whether or not stabilization will take place above *Ms*. The effect of boron on stabilization is now being studied.

**Iron Ore Recovery from Nickel Ores: Use in Steel Production**

See abstract on p. 127.

**Powder-Metallurgy Symposium**

See abstract on p. 126.

**Chromium-Nickel Powders for Production of Alloy Steel**

See abstract on p. 157.

**Influence of Composition of Steel on Resistance of Organic Coatings to Atmospheric Corrosion**

See abstract on p. 160.

**Gas-Carburizing of Low-Alloy Nickel-containing Steels**

P. PARFITT: 'Gas-Carburizing of Substitute Steels.' *Machinist*, 1953, vol. 97, May 9, pp. 745-8.

In the March 28th issue of this journal an account was given of gas-carburizing plant in operation in the works of John Fowler and Company (Leeds), Ltd. In the present article the actual process of carburizing is discussed, from the metallurgical point of view.

In common with many other users, John Fowler and Company have been obliged to accept the new low-alloy 'emergency' steels, such as En 351 (carbon 0·22 max., nickel 0·5-1·0, chromium 0·50-1·0 per cent.), and En 355 (carbon 0·20 max., nickel 1·80-2·20, chromium 1·40-1·80, molybdenum 0·15-0·25, per cent.). Details are given of the case-hardening cycle used for these steels, which is designed to obviate some of the difficulties arising from reduction in alloy content.

The low-alloy steels are found to be much more critical from the point of view of heat-treatment than the standard higher-alloy types such as En 34 (2 per cent. nickel-molybdenum) and En 39 (4¼ per cent. nickel-chromium and nickel-chromium-molybdenum), in place of which they are being used. The lower-alloy grades, when quenched direct from the carburizing treatment, show brittleness in the core, whereas both the higher-alloy steels can be direct quenched without fear of brittleness. En 351 and 355, when directly quenched, suffer from the occurrence of massive ferrite, resulting in lowered ductility, and in order to obviate this defect, a fine-grained steel is frequently specified, but there is sometimes difficulty in meeting such requirement in commercial supply.

The author of this article shows curves comparing the properties of En 34 and of En 351, the latter in conditions of varying grain size and with varying carbon content. The treatments used are designed to limit the case carbon to 0·9 per cent., which prevents exfoliation due to free cementite, and also gives the best combination of wear-resistance and ductility of case.

**Determination of Copper in High-Alloy Steels and Cast Irons**

See abstract on p. 157.

**Influence of Chromium Plating on Fatigue Limit of Nickel-Chromium-Molybdenum Steel**

See abstract on p. 136.

**Corrosion-Resistance of High-Strength Low-Alloy Steels: Correlated Data**

See abstract on p. 160.

## HEAT- AND CORROSION-RESISTING ALLOYS

**Nimonic Series of Alloys: Revision of Specifications**

Amendments have recently been issued to the three Ministry of Supply D.T.D. specifications covering, respectively, Nimonic 80, 80A, and 90. The scope of the changes is indicated below:

*D.T.D. 725: Nimonic 80*

The specification now covers extruded material, as well as material in the conditions included in the original schedule. In the section relating to testing of finished fully heat-treated blades it is specified that Brinell or Diamond Pyramid hardness tests shall be made, under conditions specified, on all finished rotor blades and on ten per cent. of finished stator blades.

A modification is also made in creep-test requirements. At 700°C. under 19 tons per sq. in., and at 750°C. under 17 tons per sq. in., creep rate during the secondary stage must not exceed 0·010 per cent. per hour. (The original figure was 0·01 per cent.).

*D.T.D. 736: Nimonic 80A.*

Modifications with regard to inclusion of extruded material, and requirements for hardness testing are as in D.T.D. 725, and for the creep test, which is at 750°C. under 17 tons per sq. in., the creep rate permissible during the secondary stage is changed from 0·01 to 0·010 per cent. per hour.

*D.T.D. 747: Nimonic 90.*

Chemical composition limits are revised, as shown below:—

| | *Original* % | *Amended* % |
|---|---|---|
| Aluminium .. .. .. | 0·8–1·8 | 0·8–2·0 |
| Titanium .. .. .. | 1·8–2·7 | 1·8–3·0 |

With regard to creep testing, the amended requirements are as follows: 'The specimen shall be heated uniformly at 815°±2½°C. and a stress of 12½ tons per square inch shall be applied, or the specimen shall be heated uniformly at 750°C.±2½°C. and a stress of 19 tons per square inch shall be applied. The temperature

at which the test is to be carried out shall be agreed between the manufacturer and the purchaser nd shall be stated on the order.'

Here also extruded material is now covered, and hardness-test and secondary-stage creep maximum requirements are in line with the modifications made in D.T.D. 725 and 736, see above.

### Precision Casting of Nimonic Alloys for Aircraft

'Precision Casting. The Precision Foundry of The Bristol Aeroplane Co., Ltd.' *Aircraft Production*, 1953, vol. 15, July, pp. 253-7.

The widespread attention which has been given during the past few years to precision investment casting by the *cire perdu* method is due to the urgent need for a process suitable for production of complicated shapes, in a large range of alloys, with a minimum of machining time. The method offers several practical advantages, among which is its use for casting to shape materials which are either unmachinable or can be machine-finished only with difficulty.

This article describes, with good illustrations, a new foundry which has recently been built and equipped by Bristol Aeroplane Company, Ltd., for the sole production of investment-cast parts. The plant is practically a self-contained manufacturing unit, including its own laboratories; it has been laid out for flow production of very large quantities of parts. This article describes the methods and equipment used at various stages of the process, and illustrates aircraft parts typical of those made by investment casting.

The new foundry is at present engaged exclusively on gas-turbine components. Parts cast in Nimonic alloys include stator blades and stator-blade segments, and swirlers for combustion chambers. These components demonstrate the chief advantage of the process, in giving increased latitude to the designer in the production of intricate shapes to close tolerances.

### Nickel-Chromium Alloy/Copper Duplex Material

*Foundry Trade Jnl.*, 1953, vol. 95, Aug. 13, p. 218.

For many years Nimonic 75 has been widely used, in sheet form, for the combustion-chamber linings in many British gas turbines, for which service the chief requirements are (1) strength to resist distortion and cracking under stresses arising from alternations in heating and cooling, and (2) good resistance to scaling. Nimonic 75 meets these requirements and, in addition, is readily fabricated and welded.

To meet the demand for a material which would have the properties characteristic of Nimonic 75, but would provide much higher heat conductivity, and thus secure more even temperature distribution in the combustion chamber, a composite metal sheet has now been developed, consisting of copper sheet clad on both sides with Nimonic 75. The metal 'sandwich', which is produced by rolling copper between sheets of the Nimonic alloy, is designated 'Nimoply'. It has many potential uses, not only in gas-turbine construction, but also in other fields of high-temperature engineering.

### Influence of Stress on Microstructure of High-Temperature Alloys

F. R. MORRAL and B. J. LAZAN: 'Metallographic Studies on N-155 Specimens Exposed to Static and Dynamic Stress at Elevated Temperatures.'
*Wright Air Development Center Tech. Rept.* 52-253, Dec., 1952; 39 pp.

This report records results obtained in one section of a series of metallographic studies on heat-resisting materials. The alloy selected was N-155 (complex nickel-chromium-cobalt-iron-base alloy containing small amounts of molybdenum, tungsten, niobium, and nitrogen).

An attempt was made to assess the effect of each of the four variables involved in the fatigue tests to which the material was subjected: time, temperature, static stress, alternating stress. Visual and macroclassifications of the fractures were made, and results of microscopical examination and microhardness data are recorded.

### Creep Properties of Chromium-Nickel and Other High-Chromium Alloys

A. H. SULLY, E. A. BRANDES and A. G. PROVAN: 'The Properties of Cast Chromium Alloys at Elevated Temperatures—

I. The Melting and Casting of Chromium-rich Alloys.
II. Some Properties of Certain Binary Chromium-rich Alloys.
III. The Creep Properties of Ternary and More Complex Chromium-base Alloys.

*Jnl. Inst. Metals*, 1952-53, vol. 81, Aug., pp. 569-84; Paper 1478.

Part I describes a furnace in which chromium and high-chromium alloys may be melted and cast in a vacuum or in an inert or reducing atmosphere. Problems connected with refractory crucibles and with investments for casting are considered. Methods for reducing the oxygen content of chromium to a low level during melting and casting are described.

Part II contains a report of compression creep tests, at 900°C., on cast chromium-rich binary alloys with iron (5, 10, 15, 20 and 30 per cent.), cobalt (10, 20 and 30 per cent.), nickel (5, 10 and 20 per cent.), palladium (6·77 per cent.), and aluminium (15 per cent.). The highest creep-resistance was found in the chromium-iron alloys containing 5-15 per cent. of iron. These alloys are homogeneous solid solutions, and were considered to constitute a promising base for further alloying, with a view to obtaining enhanced resistance to deformation at high temperatures. In some of the alloys the effect of heat-treatment on creep properties was studied. Oxidation-resistance, at 900°C., was found to be satisfactory in all the materials tested, but the alloys are brittle at room temperature.

Part III covers investigation of the chromium-iron alloys containing 5-15 per cent. of iron, with additions of tantalum, niobium, tungsten, molybdenum or vanadium. Compression creep tests were carried out at 900° and 1000°C., and tensile time-to-rupture determinations at 900°C. were made on the most promising of the ternary alloys, containing chromium, iron and

tantalum. The latter tests, however, showed wide scatter of properties, which is attributed to the effect of casting defects, in a material which is highly sensitive to notch effects. Resistance of cast chromium alloys to thermal shock is not satisfactory, and it does not appear to be possible to hot-work chromium-iron or chromium-iron-tantalum alloys.

The conclusion is drawn that it is unlikely that chromium-rich alloys can combine high creep-resistance with a degree of ductility sufficient to permit their use, with confidence, in engineering applications.

### Wear and Friction Properties of High-Nickel Alloys

R. L. JOHNSON, M. A. SWIKERT and E. E. BISSON: 'Investigation of Wear and Friction Properties under Sliding Conditions of Some Materials Suitable for Cages of Rolling-Contact Bearings.'

*Nat. Advisory Committee for Aeronautics, Report 1062*, 1952; 12 pp.

Superseding *N.A.C.A. Tech. Note 2384;* see abstract in *Nickel Bulletin*, 1952, vol. 25, No. 1; pp. 10-11.

### Exhaust Valve Materials

'Exhaust Valves: 'Notes on Materials.'

*Automobile Engineer*, 1953, vol. 43, July, p. 294.

This short note lists the properties required in exhaust valves, and emphasizes the suitability of high-nickel alloys, e.g., of the Nimonic series, for this type of service.

### Warm-Working of Austenitic Nickel-Chromium Steels

G. T. HARRIS and H. C. CHILD: 'Effect of Composition on the Warm-Working Characteristics of Austenitic Alloys.' *Jnl. Iron and Steel Inst.*, 1953, vol. 174, Aug., pp. 325-30.

It has been known for some years that the creep- and rupture-strengths of many austenitic heat-resisting materials can be improved by working the solution-treated alloy in the temperature range 600°-800°C., by hammering, stamping, rolling, etc. The solution treatment given prior to 'warm-working' is usually carried out at a temperature within 100°C. of the solidus, since it has been found that temperatures of this order are necessary to attain solution of the various carbides present, and that the maximum benefit of warm working can be obtained only when adequate solution of the precipitating carbides has been achieved.

Optimum warm-working conditions are dependent on the subsequent service or test conditions and generally the lower the service or test temperature the higher is the optimum value of the warm-working reduction. The effect of warm-working temperature is not pronounced for most alloys within the range 600°-800°C.: the lower temperatures within these limits tend to be the more suitable. It has been found that about 5 per cent. reduction in thickness, at 700°C., is a suitable treatment for austenitic materials to be used in the range 750°-800°C. and such treatment has been applied with much success to the alloy G 18B, in which, apart from improvement in creep and rupture properties, at high temperatures, proof stress at room temperature has thus been substantially raised.

In 1951 the authors described work on a range of compositions which resulted in the development of a nickel-cobalt-iron-chromium alloy strengthened by carbide-forming elements promoting precipitation hardening (*Iron and Steel Inst. Special Report 43*, pp. 67-80: *Nickel Bulletin*, 1951, vol. 24, No. 6, p. 117)*. Most of the alloys then examined were tested in the solution-treated condition, but work has since been done to determine the modification in properties obtainable by warm-working. The present paper reports the results of this investigation.

A standard treatment was adopted, consisting of:—

(1) air-cooling from 1280°C. after soaking for 2-3 minutes (but see reference in Table I to improved heat-treatment),

(2) hammering between flat tools at 700°C., to a 5 per cent. reduction in thickness.

On the alloys so treated, creep tests were made at 750°C. and, in some cases, at 800°C.

The study of the influence of warm-working was made in three sections, relating, respectively, to the effect of base composition of the alloys, the effect of carbon and of carbide-forming elements (exclusive of chromium, which is considered as a basis element), and the influence of modified heat-treatment. Full details are given of the results of each series of tests.

The conclusions drawn are that:—

'For the type of alloy under consideration, i.e., an austenitic alloy strengthened by precipitation of miscellaneous carbides, a cobalt-rich matrix is the most suitable. When carbide formers are progressively added to such an alloy of given carbon content, or when the carbon content is progressively increased with given carbide-forming elements present, the high-temperature strength of the warm-worked material passes through a maximum. This indicates the necessity for the careful balancing of carbon and carbide-forming elements. The ratio of carbon to carbide-formers required for obtaining optimum strength is somewhat less for warm-worked than for solution-treated material.

'In general, the optimum alloy for warm-working is not the most ideal for use in the solution-treated condition. As with the solution-treated alloy, there exists a 'complexity' effect, whereby the high-temperature strength properties of an alloy can be improved by increasing the number of carbide-formers present.

'The solution treatment of the alloy should be a somewhat lengthy soak (10-15 minutes) at the solidus of the alloy, followed by quenching in oil or water. The superiority of oil-quenching over normalizing is apparent for both solution-treated and warm-worked material.

'Alloys which have a low ductility in the solution-treated condition are not suited to warm-working. If ductility is high the alloy may or may not be suitable for such treatment. The carbide-forming elements niobium, vanadium, and titanium impart ductility to alloys of the type investigated, because, due to their

* Carbon 0·3, manganese 0·8, silicon 0·3, nickel 15, iron 15, chromium 19, cobalt 43, vanadium 2·8, molybdenum 2·0, niobium 1·2, per cent.

**Results of Creep Tests on a Selection of Outstanding Alloys**

Improved Solution-Treatment: Oil-quenched from 1280°C. after 10-min. soak

| Base Composition (+19-20% Cr) wt.-% | Carbon wt.-% | Carbide-forming Elements, etc., wt.-% | Stress tons/sq. in. | Temperature °C. | Time—hours to | | Elongation at Fracture % |
|---|---|---|---|---|---|---|---|
| | | | | | 1% Creep Strain | Fracture | |
| 30 Co, 40 Fe | 0·5 | 3 W, 3 Mo, 3 Nb | 16 | 750 | 150 | 167 | 2·3 |
| | | | 12 | 800 | 86 | 87 | 2·5 |
| 50 Co, 30 Fe | 0·5 | 3 W, 3 Mo, 3 Nb | 16 | 750 | 660 | 662 | 2·5 |
| | | | 12 | 800 | 338 | 339 | 1·5 |
| 64 Co, 6 Fe | 0·5 | 3 W, 3 Mo, 3 Nb | 18 | 750 | 551 | 552 | 1·6 |
| | | | 14 | 800 | 186 | 186 | 1·0 |
| 20 Ni, 20 Co, 30 Fe | 0·5 | 5·5 Nb, 3·5 Mo | 16 | 750 | 12 | 13 | 1·8 |
| | | | 12 | 800 | 24 | 79 | 9·1 |
| 20 Ni, 20 Co, 30 Fe | 0·5 | 5V, 2 Mo, 2 Nb | 16 | 750 | 110 | 218 | 11·7 |
| | | | 12 | 800 | 20 | 40 | 15·4 |
| 20 Ni, 20 Co, 30 Fe | 0·5 | 0·7 Ti, 3·2 Mo, 3·6 Nb | 16 | 750 | 68 | 69 | 3·3 |
| | | | 12 | 800 | 65 | 80 | 4·3 |
| 20 Ni, 20 Co, 30 Fe | 0·5 | 0·7 Al, 3·5 Mo, 3·9 Nb | 16 | 750 | 100 | 125 | 2·3 |
| | | | 12 | 800 | 60 | 93 | 4·5 |
| 20 Ni, 20 Co, 30 Fe | 0·27 | 3 V, 3 Mo, 3 Nb | 16 | 750 | 90 | 97 | 3·1 |
| 15 Ni, 40 Co, 15 Fe | 0·33 | 3 V, 3 Mo, 3 Nb | 18 | 750 | 200 | 324 | 9·1 |
| | | | 14 | 800 | 100 | 311 | 17·3 |
| 64 Co, 6 Fe | 0·36 | 3 V, 3 Mo, 3 Nb | 20 | 750 | 100 | 120 | 4 |
| | | | 14 | 800 | 90 | 234 | 9 |

strong carbide-forming tendency, they inhibit formation of embrittling chromium-rich carbides.

'Although warm-working is very effective in improving the rupture strength of the alloys of which the base contains a small amount of cobalt, the percentage improvement in higher-cobalt alloys is less marked.'

Some results of creep tests on a selection of outstanding alloys are shown in the Table above.

### Oxidation of Metals at High Temperatures

W. J. MOORE: 'Oxidation of Metals at High Temperatures.' *Jnl. Electrochemical Soc.*, 1953, vol. 100, July, pp. 302-13.

Data available from the literature on oxidation of pure metals from 300° to 1000°C. have been analyzed in terms of concentrations of defects in the oxide structures and defect diffusion coefficients. The original investigations on which the review is based are referred to in a *bibliography* of 61 items.

Quantitative agreement with the diffusion theory is obtained in *p*-type oxides such as cuprous oxide and nickel oxide, in which cationic vacancies arise by solution of oxygen at the oxide-oxygen interface. The parabolic oxidation rates in some *n*-type oxides, such as zinc oxide and zirconium oxide, are characterized by low activation heats and large negative activation entropies. In some of these cases anionic diffusion appears likely. In the case of zinc oxide the diffusion of radiozinc during oxidation supports the cationic diffusion mechanism. The data on linear oxidation rates fall into two classes: evidence is presented to show that one class represents reaction at the oxide-oxygen interface and the other reaction at a metal-oxygen interface.

### Structure of the Oxide Film on Nickel-Chromium Alloys

E. A. GULBRANSEN and W. R. MCMILLAN: 'Oxide Film on Nickel-Chromium Alloys.'
*Industrial and Engineering Chemistry*, 1953, vol. 45, Aug., pp. 1734-44.

The major literature on the oxidation of nickel-chromium alloys is reviewed and, on the basis of information obtained by the senior author and by other investigators, a useful tabular summary is made of surface reactions occurring on nickel-chromium alloys at elevated temperatures. These are classified in sections covering, respectively, reactions associated with direct oxidation, solid-phase reactions, formation of spinels, formation of silicates, decarburization, and vaporization. Thermodynamic evidence for the various reactions is discussed.

The experimental work reported was concerned with reflection and transmission electron-diffraction studies of the oxide films formed on a series of 80-20 nickel-chromium-base alloys. Three of the alloys selected were chosen to represent the progressive improvement in commercial 80-20 alloys which has taken place over the past 30 years (during which time the useful life of such materials has been increased by

some 600 per cent.): the compositions of the other five alloys were planned to make possible study of the effect of low manganese with low, intermediate, or high silicon, and high manganese with intermediate silicon. Life-test results, obtained by the A.S.T.M. method, are recorded for all the alloys.

The alloys were oxidized over the range 400°-950°C., for periods up to one hour at an oxygen pressure of 1 mm. of mercury. Electron-diffraction patterns indicating the crystal structure of the oxide formed on the surfaces, as a function of time and temperature of exposure, were correlated with composition and with useful life in high-temperature cyclic oxidation tests.

In 80-20 nickel-chromium alloys of low manganese (0·02-0·05 per cent.) a 300 per cent. improvement in A.S.T.M. useful life resulted from increase in silicon from 0·23 to 2·09 per cent., but the crystal structures and compositions of the oxides formed on the alloys showed no appreciable variation. Nickel oxide was formed at 400° and 500°C., chromic oxide was formed at higher temperatures, and a little spinel was found with the chromic oxide at the highest temperatures of test.

By contrast, higher manganese contents (2·30 per cent. as compared with 0·05 per cent.), in association with approximately 1 per cent. of silicon, greatly modified the crystal structure of the oxide, although the useful life proved to be independent of manganese content.

The authors conclude that there is no unique oxide composition giving good protection in cyclic tests. It is suggested that useful life is to be related not with normal oxidation processes, but rather with changes in the physical properties of the alloy-oxide interface. This is discussed with reference to the observed influence of silicon.

The paper contains a large amount of experimental data, and the authors' results and theories are correlated with those of other investigators. A *bibliography* of 30 items supports the discussion.

### 'Green Rot' in Nickel-Chromium Alloys

N. SPOONER, J. M. THOMAS and L. THOMASSEN: 'High-Temperature Corrosion in Nickel-Chromium Alloys.' *Jnl. of Metals*, 1953, vol. 5, June, p. 844: Tech. Note 164E.

Nickel-chromium and some nickel-chromium-iron alloys, when used as electrical-resistance heating elements in reducing atmospheres, sometimes suffer rapid breakdown due to a form of corrosion which, on account of its appearance, has been called 'green rot'. The reducing atmospheres producing such deterioration are most frequently those used in bright-annealing and bright-heat-treating furnaces which are kept for long periods at 1500°-1800°F. (815°-980°C.). The green rot is a preferential oxidation of the chromium in the alloy, and the resultant lowering of the chromium content of the remaining material is sometimes of the order of 10 per cent. or more.

This note makes a preliminary report on experiments carried out on 80-20 and 90-10 nickel-chromium resistance ribbons in various reducing atmospheres selected to simulate industrial conditions, e.g., moist hydrogen, and the atmosphere specified in the A.S.T.M. in 'Test for Effect of Controlled Atmospheres upon Alloys in Electric Furnaces', B 181-43. This atmosphere is of the following composition (per cent.), $H_2$ 16, CO 10, $CO_2$ 4, $CH_4$ 1, $H_2O$ 1·5-2·5, balance $N_2$.

The authors had little success in producing severe corrosion of the green-rot type in 80-20 alloys, but it was observed that the 90-10 alloys deteriorated very rapidly when placed in the bottom of narrow thermocouple protection tubes, heated in air. The effect occurred both when the wires were in porcelain tubes and when Inconel tubes were used, and subsequent investigation showed that, in such conditions, in both 80-20 and 90-10 alloys, corrosion took place much more rapidly than in the test atmospheres mentioned above.

On the basis of the observations made, the following conclusions are drawn:—

When 90-10 nickel-chromium alloys are oxidized with an abundant air supply, the oxide coating consists of both nickel and chromium oxides, and in this case thermodynamic equilibrium between the oxides is not established. When, however, the oxidized 90-10 specimens were heated at 1500°-1800°F. (815°-980°C.) inside small-diameter tubes the coating transformed to a bright metallic outer layer of nickel or nickel-rich alloy, below it was a green oxide, and underneath the oxide appeared intergranularly attacked alloy. In the narrow tubes thermodynamic equilibrium was being established according to the equation

$$NiO + Cr \text{ (in alloy)} \rightarrow Ni + Cr_2O_3$$

This reaction is favoured due to the fact that the oxygen pressure over nickel oxide is more than $10^{10}$ times that over the green chromium oxide. The oxygen initially in the tube is depleted very rapidly by oxidation of the wire and also of the tube, if it is metallic. Further experiments confirmed the accuracy of this conclusion. It appears evident that long, narrow protection tubes, whether stoppered or not, may, in certain conditions, act in the same way as sealed quartz tubes, serving as confined spaces in which oxygen is not freely replenished. Oxygen depletion can be prevented by introduction of a small amount of air into the protection tube.

The authors suggest that although other factors still remain unexplained in connexion with the occurrence of green rot, these experiments have contributed to its solution by demonstrating that the basic reaction is that of internal oxidation which occurs when the oxygen pressure is at a level sufficiently low to leave the nickel intact and preferentially oxidize the chromium.

### Precipitation-Hardening Wear- and Corrosion-Resisting Stainless Steel

N. F. MOTT: 'New Stainless Alloy Fills Long Industry Need.' *Iron Age*, 1953, vol. 171, June 18, pp. 149-53.

The author describes the development and properties of Cooper Alloy Foundry's V 2B, a low-carbon 18-8 chromium-nickel-base steel containing copper, molybdenum, silicon, and a very small amount of beryllium.

The material is covered by U.S. Patent 2,635,044. Optimum composition range is given as

| | % |
|---|---|
| Carbon | less than 0·07 |
| Chromium | 19·0–19·5 |
| Nickel | 9·75–10·25 |
| Silicon | 2·75– 3·25 |
| Manganese | 0·50– 0·75 |
| Copper | 2·0 – 2·25 |
| Molybdenum | 3·0 – 3·50 |
| Beryllium | 0·10– 0·20 |

The steel is hardenable by heat-treatment: in the quench-annealed condition it is sufficiently soft to be machined, and after machining can be precipitation-hardened by low-temperature treatment. Data on hardenability in relation to composition, obtained in developing the optimum composition, are quoted in the article.

The material is normally water-quenched from 2000°F. (1095°C.) and subsequently hardened by holding at 925°F. (495°C.) for 8 hours, followed by furnace cooling. Water quenching from higher temperatures, up to about 2300°F. (1260°C.), causes greater solubility of the beryllide hardening constituent, and hardness values up to 600 Brinell may be obtained in temper-hardened material previously quenched from these higher temperatures. Such treatment results, however, in lowered ductility, serious brittleness, and loss of machinability in the quenched condition. A table illustrates the effect of quenching temperature on final hardness values. V 2B can be produced in the cast, as well as the rolled, condition; for rolled material addition of Mischmetall is required to ensure hot workability.

The resistance of the complex steel to sulphuric, hydrochloric and phosphoric acids, and their salts, is stated to be superior to that of other precipitation-hardenable stainless steels, and better than that of Type 316 (molybdenum-containing 18-8). In nitric acid the presence of beryllium severely lowers corrosion-resistance, but the rate of attack of V 2B is still well below that of other hardenable stainless steels and the material remains practically useful in nitric-acid solutions other than those of high concentration at or near boiling temperature. The influence of the individual elements present in V 2B, on resistance to attack by the various acids, is discussed.

V 2B is claimed to be immune from over-ageing or loss of hardness after long periods of exposure at temperatures as high as 1400°F. (760°C.), a property which opens up a field for its use in high-temperature and high-pressure steam applications. Hardness values after 50 hours' exposure at various temperatures are quoted.

In the solution-annealed condition the new steel is readily weldable, using a special V 2B welding rod containing an excess of beryllium, to prevent loss by oxidation. Solution-annealing of welded components is recommended, to ensure uniformity of properties and maximum corrosion-resistance. The usual hardening treatment is then applied.

### Corrosion by Aqueous Solutions at Elevated Temperatures and Pressures

F. H. BECK and M. G. FONTANA: 'Corrosion by Aqueous Solutions at Elevated Temperatures and Pressures.' *Corrosion*, 1953, vol. 9, Aug., pp. 287-93.

Utilization of elevated temperatures and pressures in some of the industrial chemical processes recently developed has been retarded by lack of data on the resistance of metallic materials of construction to corrosion under high-pressure, high-temperature conditions. With a view to providing some of the much-needed information the Alloy Casting Institute is sponsoring research (at the Ohio State University, Columbus) on the behaviour of commercial grades of corrosion-resisting castings. This paper presents some of the test results already available.

Difficulties encountered in designing and construction of autoclaves and other test equipment for handling corrosive solutions under high-pressure, high-temperature conditions are discussed, and illustrated descriptions are given of some of the apparatus devised.

Compositions of the materials tested are given in the Table below. In the following notes the alloys of the various types are referred to, where possible, by the ACI designation used in this table.

Alloys CF-8, CF-8M and CN-7MCu were tested in the quench-annealed condition (water-quenched from 1950°F.: 1063°C.). Test periods in the autoclaves were

| Designation of Metal or Alloy | Cr % | Ni % | Mo % | Si % | C % | Cu % | Mn % | S % | N % | Fe % |
|---|---|---|---|---|---|---|---|---|---|---|
| CF-8 | 19·28 | 9·65 | Nil | 1·52 | 0·068 | Nil | 0·67 | 0·013 | 0·033 | bal. |
| CF-8M | 19·60 | 10·30 | 2·53 | 1·50 | 0·067 | — | 0·78 | 0·016 | 0·031 | bal. |
| CB-30 | 18·1 | 0·10 | — | 1·0 | 0·18 | — | 0·72 | — | 0·052 | bal. |
| CN-7MCu* | 20 | 29·7 | 2·62 | 1·15 | 0·055 | 3·84 | 0·39 | — | — | bal. |
| High-Silicon Iron† | — | — | — | 14·5 | 0·85 | — | — | — | — | bal. |
| Titanium | | | | | | | | | | |

* The Alloy Casting Institute designation covers alloys containing Cr 18-22, Ni 21-31, C 0·07 max. per cent. and varying amounts of Si, Mo and Cu. Several proprietary alloys fall within this composition range. The composition listed in the above table is for one of these alloys, known as Durimet 20.

† All tests made on Duriron

8 or 20 hours. Tests at atmospheric pressure were made in wide-mouth Erlenmeyer flasks (1000 ml. capacity), equipped with finger-type condensers. Corrosion rates reported are in many cases averages from five 48-hour periods. Results of the atmospheric-pressure tests are compared with relevant data from the literature.

Some of the major conclusions drawn are summarized below:—

*Nitric Acid*

The stainless steels showed surprisingly high rates of corrosion at slightly elevated temperatures and pressures: a relatively straight-line relationship was found between temperature and rate of attack. Alloy CF-8, exposed in 5, 20 and 65 per cent. nitric acid, showed marked increase in corrosion rate with increasing concentration of acid and rise in temperature.

There are indications that titanium and zirconium will resist corrosion at temperatures above the 345°F. (175°C.) used in the tests reported. High-silicon iron alloy shows appreciable rates of corrosion in dilute acids at temperatures above boiling point. Poor resistance to thermal shock, and brittleness, also preclude the use of this material in applications involving high temperatures and pressures. The superior resistance of CF-8 and CN-7MCu alloys, in comparison with CB-30 (straight-chromium), indicates the beneficial influence of nickel.

It is believed that nitrous acid, formed by decomposition of nitric acid under the conditions used, accounts for at least part of the increased corrosion observed in the high-pressure, high-temperature tests, as compared with results under atmospheric conditions, and this view was confirmed by marked reductions in corrosion rates of the specimens in the autoclaves when urea was added to the nitric acid or when oxygen pressure was applied. It is suggested that a better knowledge of the properties of nitric acid and of its decomposition products would be of great value in studying the inhibitor problem and in developing an alloy for contact with nitric acid under high-temperature, high-pressure conditions.

*Phosphoric Acid*

The austenitic stainless steels are, in general, satisfactorily resistant to phosphoric acid in all concentrations up to 50 per cent. acid by weight, and at all temperatures up to and including the normal boiling point of the acid. Above 50 per cent. concentration, particularly in boiling acid, these materials lose their resistance to attack. CF-8M is superior to CF-8, and the CN-7MCu type is more resistant than CF-8M, but it is not sufficiently resistant to be suitable for handling very strong acids near the boiling points.

Preliminary tests with inhibitors indicate that the addition of copper ions, as $CuHPO_4$, to phosphoric acid is effective in reducing corrosion of CF-8 alloy.

*Acetic Acid*

The CF-8 type of alloy is quite resistant to acetic acid of all concentrations and at all temperatures up to the normal boiling point of the acid: at the boiling temperature it shows borderline passivity. CF-8M and CN-7MCu types are resistant at all concentrations, at all temperatures up to and including boiling. At higher temperatures the CF-8 alloy becomes quite active, especially in glacial acetic acid. CF-8M alloy is considerably more resistant.

*Sodium Hydroxide*

CF-8 alloys are resistant to sodium hydroxide at all concentrations below 50 per cent. by weight, at all temperatures up to the boiling point. As the concentration is increased corrosion-resistance is lowered: with both CF-8 and CF-8M rise of temperature causes increased attack. The CN-7MCu alloy is much superior to the CF-8 grade in all concentrations of caustic, particularly in highly concentrated solutions at temperatures of 200°F. (93°C.) and above. At temperatures slightly above the normal boiling points of aqueous sodium hydroxide CF-8, CF-8M and CN-7MCu alloys all suffered substantially increased attack. For the CF and CFM alloys intermediate concentrations of caustic (40 and 60 per cent.) are more corrosive than 20 and 80 per cent. concentrations at similar temperatures. In 40 and 60 per cent. caustic at 425°F. (218°C.) both alloys showed severe undercutting, and a lesser degree of the same form of attack was observed at 360°F. (182°C.) in the same solutions. Preliminary tests showed CN-7MCu to be highly resistant to 20 and 40 per cent. caustic at 350°F. (177°C.).

## 'Pencilling' in Inconel Heating Electrodes in Molten Chloride Baths

H. R. COPSON: 'Corrosion of Heating Electrodes in Molten Chloride Baths.' *Jnl. Electrochemical Soc.*, 1953, vol. 100, June, pp. 257-64.

Molten salt baths are now available for almost all the heat-treatment operations required in the handling of metals and alloys: there are many types of salt and many designs of furnace for such operations. A convenient and economical means of supplying heat to the bath, particularly in the higher temperature ranges, is by means of alternating current passed through the bath between immersed electrodes, which are made from a heat-resisting alloy. The life of the electrodes varies according to the nature of the salt used and the operating conditions.

The corrosive action of the salt is usually severe at the liquid line, but in certain cases the attack is found below that line and the effect produced is a smooth wasting away, or 'pencilling', of the electrodes. Such thinning is always most pronounced on the facing surfaces of a pair of electrodes.

This paper reports a laboratory investigation of the phenomenon of pencilling, in conditions designed to reproduce commercial conditions on a small scale. The electrode material used was Inconel (nickel 77·51, chromium 14·40, iron 7·40, carbon 0·08, silicon 0·10, manganese 0·16, sulphur 0·009, copper 0·23, per cent.). The salt bath was a common neutral* chloride bath, containing 56 parts of potassium chloride and 44 parts of sodium chloride. Such baths are used in the range 1350°–1650°F. (735°–900°C.).

The results of the tests, of which full experimental details are given, led to the following conclusions:

Pencilling is an electrochemical phenomenon, and is

* Neither carburizing nor decarburizing to steel.

controlled by a protective oxide film on the electrodes. Under conditions of film breakdown the electrodes dissolve during the anodic part of the a.c. cycle. The metal ions formed plate back during the cathodic part of the cycle, as a non-adherent powder, which settles out.

No pencilling was found to occur in fresh pure salt: there was apparently sufficient oxidizing power in fresh salt to maintain a protective film on the electrodes. Alkalinity in the salt attacked the protective film and removed iron and chromium oxides from the surface, by forming chromites and ferrites. In the presence of current the film damage in alkaline salt led to pencilling. In alkaline salt pencilling increased rapidly, and at an increasing rate, with rise in current density.

Three methods of preventing pencilling were found to be effective:

(1) use of 'rectifiers' to remove alkalinity; (2) use of oxidizing agents such as carbonate or chromate, to maintain a protective film on the electrodes, and (3) use of a low current density.

**Thermal Conductivity of Nickel and Nickel Alloys: Summary**

See abstract on p. 128.

**Addition of Rare-Earth Metals and Compounds to Stainless Steel**

C. B. POST and H. O. BEAVER: 'Use of Rare-Earth Metals and Compounds in Stainless Steel Melting.' *Blast Furnace and Steel Plant*, 1953, vol. 41, June, pp. 627-34, 645: see also summary in *Iron Age*, 1953, vol. 171, June 4, pp. 148-9.

This paper is a sequel to that published by the same authors in 1951 (*Jnl. of Metals*, 1951, vol. 3, Nov., pp. 973-77B: *Nickel Bulletin*, 1952, vol. 25, No. 1, pp. 21-2). Most of the data then presented were obtained on high-alloy stainless steels melted in high-frequency furnaces, and the experiments had been made primarily with a view to improving the hot-workability of such materials. It was known at that time that in lower-alloy stainless steels, melted in electric arc furnaces (e.g., Types 308, 316 and 310), smaller amounts of Mischmetall than those necessary in the h.f. steels sufficed to effect improvement in hot-working quality. Such steels are not, in general, hot-short, and the effect of Mischmetall will be primarily reflected in improved yield, for example, of prepared billets, over a large number of heats. Study of the influence of such additions, on the electric-arc furnace steels, has been carried out since that time and the results have confirmed early observations.

It has also been established that stainless steels such as 308, 316 and 310 are in some cases improved by ladle additions of mixtures of the rare-earth oxides with suitable reducing agents, provided that the melting practice is such that ductile metal is obtained without such addition. In this case the residual cerium and lanthanum contents are very small or, in some cases, there is no such residual. Addition of mixtures of rare-earth oxides does not improve hot-workability of essentially hot-short or difficult-to-work stainless steels.

The observation of these latter effects led to the assumption that the improvement in hot-working properties of the inherently ductile grades of stainless steel by means of rare-earth oxide treatments might be due to a mechanism different from that involved with Mischmetall additions. Work in the authors' laboratories since 1951 has therefore been concentrated on investigation of the mechanism of the two types of reaction. This paper reports some preliminary results.

As an introduction to the summary of experimental data, a review is made of the main factors which influence hot-workability of stainless-steel ingots:—(*a*) hot-shortness, (*b*) fluidity, (*c*) ingot structure. Reference is made to the cone test developed for assessment of hot-working properties, and some results typical of tests on various grades of stainless steel are quoted.

The conclusions, to date, with regard to Mischmetall additions are as follows:—

(1) Mischmetall additions to high-alloy stainless steels which are inherently hot-short confer ductility. The authors' experience is that these types require a minimum cerium+lanthanum content of 0·02 per cent.

(2) Mischmetall additions to lower-alloy austenitic stainless steels which are inherently ductile (e.g., 316, 308, 310, etc.) may result in improvement of hot-workability as judged by yield of prepared billets or slabs.

(3) The relative amounts of Mischmetall required for treatment of the different grades of stainless steel suggest that cerium+lanthanum not only counteracts hot-shortness but simultaneously improves fluidity and the ingot structure.

Cerium-fluoride additions, to ladle and/or moulds, have also been found to improve the ingot surface and hot-workability of austenitic stainless steels containing titanium. The amounts are of the order of 2-3 lb. per ton to the ladle and/or 2 oz. per 1000 lb. metal in the mould.

Investigations of the influence of Mischmetall on the sulphur content of stainless steels were made on an h.f. heat of the following composition:—carbon 0·046, manganese 0·89, silicon 1·12, chromium 20·23, nickel 28·83, copper 3·69, molybdenum 2·50 per cent. Cone tests indicated that the mere lowering of sulphur in this grade of steel does not ensure hot-workability or lead to the absence of hot shortness, but the presence of residual cerium+lanthanum of 0·08-0·17 per cent. ensures the absence of hot-shortness over a considerable range of sulphur content.

With regard to the mechanism of the effect of Mischmetall, the authors' results to date confirm early indications that a strongly deoxidizing effect is a major factor. There is also some evidence that cerium and lanthanum have an effect in overcoming the injurious influence of 'tramp' elements such as lead, tin and silver. It is urged that, in the interest of use of minimal amounts of Mischmetall and of preventing excessive formation of non-metallics, prior deoxidation of the metal, in the ladle, by some strong conventional

deoxidizer such as calcium-silicon, is desirable. Two other factors in preventing formation of non-metallics in Mischmetall-treated steels are the use of tapping temperatures as low as is consistent with other requirements, and protection from the atmosphere, to inhibit oxygen take-up.

### The Delta-Ferrite Phase in Nickel-Chromium Austenitic Steels

J. BROWN, D. CLARK and W. D. CLARK: 'Stability of the So-called Delta-Ferrite Phase in Austenitic Steels.' *Jnl. Iron and Steel Inst.*, 1953, vol. 174, Aug., pp. 316-17.

The existence of a proportion of a body-centred cubic ferromagnetic phase in many austenitic steels at room temperature is well known, and, as it has been commonly supposed that this phase remains relatively unchanged from room temperature up to the solidus, it is frequently designated δ-ferrite, by analogy with the δ-phase in pure iron.

In many austenitic steels ferromagnetism may be induced or increased by cold-work, due to production of a body-centred phase by transformation of metastable austenite. It has been assumed that, on heating such cold-worked steels to a temperature sufficiently high to transform the martensite back to austenite, the effects of the cold-working would be completely removed.

The writers of this note present evidence to refute both these presumptions.

In experiments with various complex austenitic steels they have found that the body-centred phase in duplex steels is not stable at temperatures in the region of 900°C., but transforms to a face-centred cubic phase. It is known that annealing such steels at temperatures above about 1000°C. causes an increase in the proportion of ferrite found after the steel has been air-cooled or quenched, and the ferrite, when microscopically examined, shows no sign of having been formed from the austenite matrix during cooling. It is therefore concluded that when the steel is hot two f.c.c. phases are present, one of which transforms, on cooling, to b.c.c. ferrite, whilst the other is stable.

Effects produced on cold-working and annealing are demonstrated by reference to observations on plain 18-8 chromium-nickel and 18-8-Ti steels. In cold-worked specimens a high proportion of b.c.c. 'martensite' was present at temperatures up to 600°C., but at 650°C. this disappeared and the austenite lines became stronger. On cooling, the b.c.c. lines re-appeared. The effect of cold-working was not completely removed until the specimens were heated at 850°C. The authors suggest an explanation for the reversible transformation of the martensite, which is unexpected in view of the presumption that the f.c.c. phase to which it transforms would be identical with the original austenite, and therefore completely stable on cooling. They consider it possible that the cold-working ejects carbides from the lattice, and since these carbides do not re-dissolve at 650°C. the f.c.c. phase formed from the martensite at that temperature has a composition different from that of the original austenite, and less stable.

### Structural Effects in Acid Corrosion of Chromium-Nickel-Molybdenum Steels

H. T. SHIRLEY: 'Microstructural Characteristics of Acid Corrosion in 18% Cr, 8-14% Ni, 3% Mo Steels.' *Jnl. Iron and Steel Inst.*, 1953, vol. 174, July, pp. 242-9.

Microstructural features observed in titanium- and niobium-stabilized steels corroded by nitric acid were described by the author in an earlier paper (*ibid.*, 1952, vol. 170, pp. 111-18; *Nickel Bulletin*, 1952, vol. 25, No. 4, p. 115). Particular attention was directed to effects of complex heat-treatments introduced during double welding. It was concluded that acceleration of corrosion was by grain-boundary attack, and was related to chromium impoverishment by carbide precipitation. Boiling 70 per cent. nitric acid proved to be a much more drastic corrodent than the standard sulphuric-acid/copper-sulphate solution for detection of susceptibility to intergranular corrosion, and it was observed that steels effectively stabilized by titanium with regard to the standard test, and against most service conditions, were markedly inferior to niobium-stabilized steels in resistance to nitric acid. Delta boundaries in the titanium-stabilized steels were particularly susceptible to attack.

In later work on titanium- and niobium-stabilized steels (18/8-14 chromium-nickel types) the carbon content was varied, and tests were made in solutions of acetic, hydrochloric, and sulphuric acids, as well as in boiling 65 per cent. nitric acid. In this series only the nitric-acid solution produced corrosion in steels satisfactorily stabilized against the acidified copper-sulphate reagent. As in the earlier tests, corrosion was particularly severe round the delta boundaries in the titanium-stabilized steels. (*ibid.*, 1952, vol. 172, pp. 377-80; *Nickel Bulletin*, 1953, vol. 26, No. 1, p. 23).

Neither group of studies permitted investigation of the influence of sigma to be studied, since none of the steels used showed any indication of sigma formation even after heat-treatments of 8 hours at 750°C., or 4 hours at 850°C. In view of the fact that molybdenum-bearing steels are more prone to development of sigma, such types were selected for the further research which is the subject of the present paper. The steels used were of the following types:

| | |
|---|---|
| 18–9–3 Cr–Ni–Mo | Ti-stabilized |
| 18–8–3 Cr–Ni–Mo | |
| 17–11–2½ Cr–Ni–Mo | |
| 17–12–2½ Cr–Ni–Mo | |
| 17–14–2½ Cr–Ni–Mo | |
| 17–12–2½ Cr–Ni–Mo | Nb-stabilized |
| 17–8–3 Cr–Ni–Mo | |

Tests were made in nitric-, sulphuric-, and hydrochloric-acid solutions, after various heat-treatments, and structural changes produced by such treatments were correlated with the resistance of the steels to corrosion attack.

In boiling concentrated nitric acid the most damaging attack is intergranular. In the 18-8-3 chromium-nickel-molybdenum titanium-stabilized steel this deterioration was greatly accelerated by prior treatment at

650°C.: the effect is attributed to chromium impoverishment by carbide precipitation at the austenite boundaries, and it is possible that titanium is also deleterious. In the nitric-acid tests the only one of the steels showing immunity from the accelerating effects of heat-treatment was the 17-12-2½ chromium-nickel-molybdenum steel stabilized with niobium.

Sigma phase in the 18-8-3 chromium-nickel-molybdenum titanium-stabilized steel is still higher in chromium and molybdenum content than the delta from which it is formed, and it has a very high resistance to acid attack, but the formation of sigma correspondingly impoverishes the surrounding material, and local depletion effects in this transformed material are believed to account for higher attack by nitric acid in these regions. This effect is not, however, sufficiently rapid or continuous to be comparable with the intergranular effect resulting from treatment at 650°C. In sulphuric-acid solutions molybdenum depletion of the transformed regions has probably a major effect in lowering resistance to corrosion.

Virtual elimination of delta in 17-12-2½ chromium-nickel-molybdenum titanium-stabilized steel produced conditions less favourable to the damaging effect of the 650°C. treatment, but increased the danger from sigma formation, by making this essentially a grain-boundary precipitation from the austenite, with sufficient continuity to give rise to severe attack by nitric acid after treatment at 750°-850°C. In this steel resistance to sulphuric acid was unaffected by heat-treatment.

Behaviour in sulphuric and hydrochloric acids was found to depend on differences in phase behaviour, rather than on boundary effects. The higher chromium and molybdenum and the lower nickel contents of delta, compared with those of austenite, in 18-8-3 chromium - nickel - molybdenum titanium - stabilized steel, may lead to variation in degree, and even to reversal, of selective attack, according to variation in the concentration of the reagent and its temperature. Lowering of molybdenum content in transformed delta, as the result of formation of sigma, is considered to be the major cause of increased attack of sulphuric acid on this steel.

### Corrosion of Austenitic Chromium-Nickel Steels in Oxidizing Solutions

'Corrosion of 18-8 in Oxidizing Solutions.' *Metal Progress*, 1953, vol. 64, July, pp. 124, 126.

Abstract based on paper by M. M. KURTEPOV and G. V. AKIMOV (*Doklady Akad. Nauk S.S.S.R.*, 1952, vol. 87, p. 1005).

In a previous investigation of the influence of nitric acid containing oxidizing agents, on the corrosion of 18-8 chromium-nickel steel, the authors had found that, contrary to the conclusion of others, such steels showed poor resistance to corrosion in oxidizing solutions. The paper abstracted in Metal Progress gives results of a study of the influence of additions of the oxidizing agents $K_2Cr_2O_7$, $NH_4VO_3$, and $KIO_3$, on the electrode potentials and kinetics of the electrode processes on stainless steels. Typical Russian 18-8 steels, containing niobium or molybdenum + niobium, were used. The main observations are summarized in the abstract.

### Colour and Reflecting Properties of Stainless Steels and Other Materials

F. K. BLOOM: ''Color' and Reflectance of Stainless Steels.' *Metal Progress*, 1953, vol. 63, June, pp. 67-72.

This paper reports results of an investigation, in the laboratories of Armco Steel Corporation, of the reasons for the difference in appearance between stainless steels and other metals, the influence of variations in composition within the stainless-steel ranges, and the effect, on appearance, of various methods of polishing the surface.

Consideration of the nature of the physical factors affecting reflectance and colour indicates that, broadly speaking, the differences in appearance of similarly polished surfaces of decorative metals are due primarily to variations in their absolute reflectance. Silver is an unusual metal, in that it reflects nearly 95 per cent. of visible light, whereas most other metals, including stainless steels, may absorb up to about 40 per cent. of the light. Most metals tend to reflect the red end of the spectrum better than the blue end, and, depending on the extent of this difference, some are characterized by a faintly yellowish hue. The plain-chromium steels have no visible hue and are slightly lower in reflectance than the chromium-nickel types: the latter have an extremely faint tan-to-yellow hue. Variations in alloy content of the stainless steels affect their colour only slightly.

The investigation made in the author's laboratory was based on a physical comparison of the colour of the different metals, by establishing the amount of reflection obtained at various wave lengths in the visible spectrum. Experimental methods are described. The detailed results, presented in curves and tables, cover the characteristics of the following types of materials: straight-chromium steels of six types; austenitic chromium-nickel steels of six compositions; stainless chromium-manganese-copper steels; Armco ingot iron; silver plate and sterling silver; nickel silver; chromium plate; aluminium; Monel; nickel; copper.

The apparent brightness of a surface depends on its absolute reflectance and also on the nature of the surface texture and character of the illumination by which it is viewed. Where the illumination includes some light from a point source, a surface finish which catches and disperses some of the special illumination has an increased apparent brightness. For the purpose of increasing apparent brightness, grit blasting followed by electropolishing is likely to give specially good effects. Embossed designs are also effective in brightening the appearance of articles and providing contrasts.

The finishing tests on stainless steels suggest that polishing methods such as buffing, although increasing the lustre and image-forming characteristics of a surface, may actually lower absolute reflectance slightly, and increase the depth of the yellow hue. Where highest possible reflectance is desired electropolishing appears to offer advantages.

Whereas the absolute reflectance of stainless steels is an inherent property, similar to and related to electrical conductance, apparent reflectance is susceptible to variation over a considerable range, by modification in methods of surface preparation.

**Simplification of Stainless Steel Specifications**

'Symposium on the Simplification of Stainless Steel Specifications.' *Trans. Instn. Chemical Engineers*, 1953, vol. 30, pp. 151-75.

Report of papers and discussions presented at the symposium organized by the Institution of Chemical Engineers in April, 1952. The scope of the individual contributions is indicated below:—

J. L. SWEETEN: 'The Simplification of Specifications for Stainless Steels,' pp. 152-5.

This introductory paper outlines the aim of the symposium, which was to 'crystallize opinion on the specifications for stainless steel in most common use in the chemical, petroleum, and allied industries'.

Reference is made to British Standard specifications covering such materials, and, following criticism of some of these schedules, proposals are made for revised specifications for (1) 18-8 and 18-8-stabilized steels and (2) 13 per cent chromium and 18-8-Mo steels. In a further table the proposed composition groups are arranged according to the physical shapes for which they would be used (bars, plates, castings, pipe, etc.). Specific suggestions are made for the withdrawal of certain B.S. specifications, and for the simplification and modification of others, with particular reference to the needs of the petroleum industry, the tonnage requirements of which may soon exceed those of any other process industry.

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E. SMITH: 'The View of the Steelmaker,' p. 156.

In this paper the opinion was expressed that existing B.S. specifications satisfactorily meet the essential requirements of the industries using them, and that the tentative proposals put forward in the previous paper did not constitute any improvement on existing schedules. The suggestion was made that the B.S.I. could usefully form a technical committee consisting of plant users, plant fabricators, and suppliers of materials (not necessarily confined to stainless steel), and that such a committee could, with advantage to the industries concerned, consider general problems as and when arising, and offer ad hoc advice. It was urged that such a course would be likely to provide more practical service than rigid reliance upon specifications.

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M. M. HALLETT: 'The Foundryman's Point of View,' pp. 157-8.

The proposals made for modification of existing specifications are evaluated on the basis of the ease or difficulty of foundry production of the respective types. On balance, the author concludes that the proposed specifications do not show any advantage over existing B.S. grading and classification.

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J W. JENKIN: 'The View of the Semi-Finished Product Maker,' pp. 159-61.

While conceding that there is much advantage in having the minimum number of varieties in raw materials, from the points of view of the primary producer, the maker of intermediate products, and the final user, the argument is advanced that any co-ordination of stainless-steel specifications on a composition basis is justified only after full consideration of the following factors:—

(1) the technique of manufacture at every stage, of the various forms and shapes in which the material may finally be used; (2) the importance of ensuring the maximum number of satisfactory products for the minimum number of varieties in composition; (3) the need for limits to be set, by proof that detriment would follow if they were exceeded; (4) the prejudice against change from established practice without most cogent reasons; (5) the fact that existing compositions have been built upon many years of experience and that any change would have to be gradual, in order to utilize existing stocks; and (6) the necessity to avoid changes which would worsen the supply position.

From the standpoint of these factors, detailed comment is made on specifications proposed to cover martensitic, ferritic, and austenitic grades of stainless steel.

(Discussion on these papers, pp. 161-4.)

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J. F. LANCASTER: 'The Fabricator's Standpoint,' pp. 165-7.

This paper deals with the proposed list of specifications from the point of view of the weldability, cold- and hot-working, and machinability of the steels covered. The author was of the opinion that, generally speaking, the draft proposals represented no great advance on existing specifications such as B.S. En 56, 57, and 58, except for the inclusion of the stainless irons.

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F. H. KEATING and W. D. CLARKE: 'The View of the User,' pp. 168-9.

The authors emphasize that standardization, by its very nature, is a compromise to provide the greatest possible benefit for the largest possible number of people concerned in manufacture, handling and use of materials. Some sacrifice of perfection to expediency is inevitable, and the ideal is attained when sacrifices are shared, in reasonable proportion, by all interested parties. The specific points singled out for discussion, bearing in mind the need for practical compromise, are: (1) the carbon content of the 18-8-Ti wrought steel; (2) the advisability of inclusion, in revised British specifications, of the extra-low-carbon types; (3) modifications which have already been introduced in molybdenum-bearing austenitic steels to inhibit sigma formation; (4) stabilization by titanium or niobium. In connection with stabilization, the authors consider that 'unstabilized molybdenum-free steels have no use in chemical plant,' since frequently the exact duty for which a given steel is intended is not stated by the purchaser, and unstabilized steels do, in some circumstances, suffer disintegration. In selecting materials for expensive plant which is to run for many years no risk should be taken which can be so easily avoided.

With regard to existing B.S. specifications, the view is expressed that schedules in B.S. 970 have ranges of composition which are too wide for the convenience

of users, and leave too much to be written into orders. The B.S. 1501 series is considered to come much closer to practical requirements, and would, with some amendments, be acceptable to the majority of users in the chemical industry.

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G. A. DUMMETT: 'Substitute Stainless Steels,' pp.170-2.

Alternative compositions which have been, or might be, considered in order partially to obviate use of nickel and molybdenum in stainless steels are discussed, in relation to the influence of reduction in these elements on corrosion-resistance, forming properties, etc. The subject is dealt with in three groups: ferritic, austenitic, and ferritic-austenitic types. General experience confirms that there is no direct substitute for the austenitic stainless steels for all forms and duties, although in some cases it is possible to use ferritic steels and steels of ferritic/austenitic types. Difficulties confronting the steelmaker in large-scale substitution are emphasized.

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Discussion on the second group of papers, pp. 172-4.

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E. T. HOBLYN: 'Resumé of Papers and Discussions,' pp. 174-5.

The main points from the respective papers are brought out in this resumé, and it is suggested that in view of the obvious importance of the subject of standardization, a report on the symposium should be submitted to the British Standards Institution, accompanied by a recommendation that the B.S.I. should arrange for interchange of views among all interested parties, with the aim of—

(*a*) revision, co-ordination and condensation of existing British Standards for stainless steels, and preparation of new standards to fill any important gaps thereby created;

(*b*) preparation of a set of parallel specifications for substitute stainless steels, for present use, and as an insurance policy in case of emergency.

### Oxalic-Acid Electrolytic-Etch Test for Stainless Steels

M. A. STREICHER: 'Screening Stainless Steels from the 240-hr. Nitric Acid Test by Electrolytic Etching in Oxalic Acid.' *A.S.T.M. Bull.*, 1953, No. 191, July, pp. 58-9.

Discussion, by D. J. CARNEY, of paper published *ibid.*, 1953, No. 188, Feb., pp. 35-8: see *Nickel Bulletin*, 1953, vol. 26, No. 5, p. 88.

D. J. CARNEY described an electrolytic weight-loss test in oxalic acid which has been developed in the laboratories of United States Steel Corporation, to give, in a short time, quantitative results comparable with those obtained by the longer 65 per cent. boiling nitric-acid test. Data already available indicate a good degree of correlation between the two. The electrolytic weight-loss test has at present been tried on only Type 304 extra-low-carbon chromium-nickel steel, and the immediate objective of further work is to test other grades of stainless steel. There appears to be some possibility that if consistent agreement can be established, the oxalic-acid weight-loss procedure may replace the nitric-acid procedure.

In reply, STREICHER gave some information on further work which has been carried out in the Du Pont laboratories on the relationship between oxalic-acid etch structures and nitric-acid corrosion rates. Structures produced by this procedure on austenitic steels have been found to be divisible into three categories: 'step', 'dual', and 'ditch'.

By evaluation of the structures in accordance with these criteria the steels can be sorted into two groups: those which drop grains in the nitric-acid test, and those which do not do so. Experience to date shows that the percentage of steels which can be eliminated from nitric-acid testing by oxalic-acid etching is 75-90 per cent. for Types 304 and 316 and about 50 per cent. for sensitized 304L.

Some comments were also made by Streicher on apparent limitations in the utility of the oxalic-acid electrolytic weight-loss test at its present stage of development.

### Nickel-Chromium Stainless Steel in Structural Applications

A. G. DEAN: 'Structural Applications of Stainless Steel.' *Engineer*, 1953, vol. 195, June 19 and 26, pp. 853-5, 882-4.

Serious consideration of stainless steel as a structural material started about 1930, first for airframes, and later for railway coaches and other applications. Many problems of fabrication and design arose in using this type of material for such purposes, and a considerable amount of information has been built up during the past twenty years. This article reviews the subject on the basis of the experience of The Budd Company, which has been a pioneer in this field of application: it is concerned chiefly with the use of stainless steel in railway coaches, in which that Company has specialized.

It is stated that during the past twenty years about 1950 railway coaches have been built for use in America: for service in Europe some 400 have been built or are on order. About 23,000 trailer cars of stainless steel have been constructed over the same period.

Design problems involved in using this material for such purposes are discussed, methods of stress analysis applicable to coach structures are considered, and measures adopted to ensure maximum crash-resistance are mentioned. The value of the corrosion-resisting characteristics of stainless steel, in structural applications, are emphasized, and service behaviour in railway service is recorded. Finally, the author calls attention to the mechanical limitations of stainless steel, and gives warning against its use in applications for which its properties are not suitable. The data now available justify the claim that where long life is required in a structure, and particularly in cases where light weight is important, reduced maintenance cost can justify considerable increase in first cost of a material. Stainless steel has given striking proof of this argument.

Coaches have been operating in the corrosive atmosphere common to all railways for nearly twenty years, without indication of deterioration, although most of the exterior sheathing is only 0·020 in. thick. It is urged that there are many other applications in which the choice of stainless-steel construction would be an economic proposition.

**Nickel-containing Materials in Surgical Apparatus**

'60% Cost Saving with Precision Casting.' *Nickel Topics*, 1953, vol. 6, No. 6, p. 6.

An illustrated description is given of improved suturing apparatus now being widely used in abdominal surgery. This equipment speeds up such operations by eliminating the use of needles, needle holders and catgut. The unit permits a surgeon to insert parallel rows of up to 23 pairs of staples into the tissues during an operation, to close the edges of incisions: it may also be used to isolate certain parts of organs before they are cut open.

The small staples are made of nickel silver (20 per cent. nickel), which resists corrosion from chemical compounds in the body, and does not create toxic effects. The staples remain in place until 8-10 days after the operation, when they are released from the healed tissues and eliminated from the body. Lugs used to force the staples out of the upper jaw of the apparatus and through the tissues are made from nickel-chromium stainless steel. They were formerly milled from bar stock, but the introduction of precision casting has cut cost of production by some 60 per cent.

**Manufacture of Nickel Alloy Coil Springs for Watches**

See abstract on p. 141.

**Nickel Equipment in Salt-Producing Plant**

See abstract on p. 130.

**Wrought Corrosion-Resisting Nickel-Molybdenum Alloys**

'Wrought Nickel-Molybdenum Alloys.' *Engineering*, 1953, vol. 176, Aug. 21, p. 248.

Investigations carried out some years ago, in both U.S.A. and Germany, demonstrated that the addition of molybdenum to nickel, or to nickel alloyed with small percentages of iron, produced materials which are strongly resistant to corrosion by hydrochloric and phosphoric acids, and, in certain conditions, by sulphuric acid. Cast forms of such alloys have been available in England for some time, but a corresponding wrought alloy has only recently become available.

An alloy of this type is being produced (by Messrs. Henry Wiggin and Company, Ltd.), under the name of Corronel B, the nominal composition of which is nickel 66, molybdenum 28, iron 6, per cent. Previous study of the alloys, supplemented by more recent laboratory investigations, has shown that corrosion-resistance of these alloys at about the 30 per cent. molybdenum level is unimpaired by the addition of up to 10 per cent. of iron. Corronel B can be satisfactorily worked, although it offers somewhat greater resistance to deformation than mild or low-alloy steels.

**Welding of Stainless Steels**

H. THIELSCH: 'Welding the Stainless Steels.' *Materials and Methods*, 1953, vol. 37, May, pp. 115-30: *Materials and Methods Manual No.* 94.

This summary of information is based on a comprehensive report prepared by the author while he was Technical Assistant in the Welding Research Council. It contains practical recommendations on materials and technique, classified in sections covering, respectively,

composition and properties of electrodes for welding stainless steels of austenitic and ferritic grades;

the technique of welding austenitic stainless steels (including discussion of the problem of carbide precipitation, formation of sigma phase, hot cracking of weld metal, pre-welding treatments, and annealing and stress-relieving after welding),

the technique of welding fully and partially martensitic stainless steels, and the choice of suitable austenitic and martensitic electrodes,

the technique of welding the ferritic stainless steels (including use of austenitic and of ferritic electrodes),

welding procedure, joint designs and joint preparation (including discussion of shielded metal-arc, submerged-arc, atomic-hydrogen, inert-gas, oxyacetylene, and resistance welding; brazing and soldering)

A list of detailed correlated review articles on welding of stainless steels, which have been sponsored by the Literature Advisory Committee of the Welding Research Council, is appended to the article.

**Cone Arc-Welding Process**

P. PATRIARCA and G. M. SLAUGHTER: 'Cone Arc Welding.' *Welding Jnl.*, 1953, vol. 32, July, pp. 597-602.

During recent years there has been a growing interest in the application of miniature heat exchangers of the tube-to-header type design, for elevated-temperature service in the presence of cooling media which constitute severely corrosive environments. Fabrication of these assemblies, consisting of a multitude of thin-walled tubes of small diameter welded into a single header, proved difficult when conventional welding processes were used, and it has been found necessary to develop a new semi-automatic process.

The method devised, which is described in this paper, is known as the Cone Arc Process. The basic principle is the rotation of the arc round the header hole at high speeds, giving uniform distribution of the energy required to make a peripheral weld of a tube into a header sheet. Equipment and technique are described, and typical welds made by the process are illustrated.

The author reports an evaluation of the practicability of the process, using Type 316 austenitic stainless steel tubing, 0·100-in. o.d., 0·010-in. wall thickness, and Type 316 steel sheet 0·125-in. thick, from which experimental heaters were machined. The work was carried out in the Welding Section of the Metallurgy Division, Oak Ridge National Laboratory.

The results of the investigation showed that the

major variables (arc current and arc time) are not critical within reasonable limits, and it was found possible, by correct selection of welding conditions, to obtain penetrations of the order of two to three times the thickness of the tube wall without objectionable reduction of the inside diameter of the tube. Various combinations of Cone Arc welding conditions found to give specially good results were used in evaluating the consistency and reliability of the process, which is considered fully satisfactory for the purpose involved.

### Resistance Welding of Stainless-Steel Aircraft Components

D. O. SAMUELSON: 'Resistance Welding Applications in Aircraft Stainless Steels.' *Welding Jnl.*, 1953, vol. 32, July, pp. 603-11.

Detailed information is given on specific applications of spot and seam welding in aircraft parts of nickel-chromium stainless steels, high-nickel alloys and some low-alloy constructional steels. The methods and equipment described relate to practice in the welding shops of Solar Aircraft Company, San Diego, California. The article is well illustrated.

### Influence of Copper in Nickel-Chromium Stainless-Steel Weld Metal

R. P. WENTWORTH: 'Copper in Type 347 Weld Metal.' *Welding Jnl.*, 1953, vol. 32, July, Suppl., pp. 347S-51S.

It has been suggested in the literature that the addition of small percentages of copper to stainless-steel weld metal tends to inhibit formation of sigma. Relevant statements are quoted.

The author suggests that if copper does indeed exert such an influence, the mechanism of action must be one of the following, or a combination of two or all three of them: (1) reduction of the original amount of ferrite present; (2) reduction in the rate of sigma formation; (3) reduction of the inherent brittleness of the sigma particles. The first effect will result if copper is an austenite-forming element, but since, to ensure freedom from weld-cracking, it is desirable to maintain a certain level of ferrite, this would not be a practical solution to the question. The second mechanism appeared to be a reasonable one, and was, therefore, investigated by the present author, using hardness and magnetic tests. As a means of examining the third possibility, free-bend tests were made.

In order to ascertain the effects of varying amounts of copper and ferrite, nine electrodes were prepared, giving the following levels of composition:—copper 0, 2, and 4 per cent.; ferrite 0, 4, and 9 per cent. Later, four additional electrodes were made, having a higher silicon content: these had, respectively, copper 0 and 2·5 per cent., and ferrite 4 and 9 per cent.

The conclusions drawn from the experimental data recorded are that:—

(1) Tests on specimens treated at two sigmatizing temperatures 1200° and 1500°F. (648° and 815°C.) show that copper does *not* retard rate of sigma formation, even in the presence of higher-than-normal silicon.
(2) Copper does *not* appear to alter the microstructure of as-welded or heat-treated weld metal: sigma-containing structures of the heat-treated specimens are illustrated.
(3) Bend tests revealed no lessening of the brittleness of the sigma phase in copper-containing specimens.

It is suggested that the above conclusions should be treated as preliminary, since possibly longer times of treatment might reveal some benefit from copper in relation to the sigma problem. It is also thought desirable that the influence of heat-treatment prior to sigmatizing should be investigated.

### Spectrographic Analysis of Nickel-Chromium Stainless Steel

N. E. GORDON, R. M. JACOBS and M. C. RICKEL: 'Spectrographic Analysis of 18-8 Stainless Steel for Cobalt, Manganese and Stabilizing Elements.' *Analytical Chemistry*, 1953, vol. 25, July, pp. 1031-4.

The method described was developed to obviate expenditure of time required in wet chemical procedures for estimation of niobium, molybdenum, tantalum, cobalt, titanium, and manganese in austenitic nickel-chromium steel. A salt technique is used, with an alternating current arc excitation. Precision of 10 per cent. is obtained. The method is particularly suitable where samples of diversified shape, size, and metallurgical history are involved. Standards required can readily be synthesized from the respective pure metals and analytical grade reagents. Values obtained by the spectrographic method, in typical determinations of the elements mentioned, are compared with values given by standard chemical analysis procedures.

### Determination of Copper in High-Alloy Steels and Cast Irons

BRIT. IRON AND STEEL RESEARCH ASSOCN., METHODS OF ANALYSIS COMMITTEE. 'Determination of Copper in Iron and Steel.' *Jnl. Iron and Steel Inst.*, 1953, vol. 174, Aug., pp. 335-8.

Two methods for determination of copper are at present in existence as British Standards: they cover a range of compositions in the permanent-magnet and low-alloy classes of steels (B.S. 1121, Parts 5 and 14). These procedures have been under review and the investigation reported in this paper was carried out to extend and modify the methods in such a way as to make them applicable to all classes of steel. Full details of the improved procedure are given, and determinations of copper in typical high-alloy steels and cast irons are shown, to demonstrate its application.

### Chromium-Nickel Powders for Production of Alloy Steel

J. D. SHAW, W. V. KNOPP and B. A. GRUBER: 'Chromium-Nickel Alloy Steel Sinterings: an Investigation of Physical Properties.' *Precision Metal Moulding*, 1953, vol. 11, Mar., pp. 42-5, 73-6.

The investigation reported covers a study of the possibility of employing chromium-nickel alloy powders

produced by the Hydride process, in production of alloy steels. By this process fine, pure primary metal oxides, blended in correct proportions to form the required alloy, are thermally reduced by calcium hydride. The resulting powders, which are of fine particle size, have a low oxide content, and are also characterized by a relatively porous structure containing particles of irregular shape. The powders are easily moulded, and blend readily with conventional iron powders.

In the experiments described in this paper 80-20, 50-50, and 20-80 chromium-nickel alloy powders were used. These were mixed, in amounts of 5, 10 and 20 per cent. alloy, with electrolytic iron powder (with or without 1 per cent. of graphite) and the properties of compacts prepared from these mixtures were determined.

The results of the first group of tests (chromium-nickel-iron) are shown in Table 1, below:

**Table I**

**Electrolytic Iron Powder + Chromium-Nickel Powders**

Compacting pressure: 50 t.s.i. Sintering conditions: 1100°C. for one hour in hydrogen.

| | Contained Alloy | | |
|---|---|---|---|
| | 5% | 1% | 20% |
| 80 Cr/20 Ni | | | |
| Sintered density, g./c.c. | 6·93 | 6·75 | 6·60 |
| Tensile strength, p.s.i. | 42,900 | 54,800 | 61,100 |
| t.s.i. | 19·15 | 24·45 | 27·30 |
| Elongation, % on 1 in. | 7·2 | 2·5 | 2·0 |
| Rockwell hardness | B 32 | B 54 | B 63 |
| 50 Cr/50 Ni | | | |
| Sintered density, g./c.c. | 6·98 | 6·82 | 6·63 |
| Tensile strength, p.s.i. | 29,100 | 39,100 | 46,200 |
| t.s.i. | 13·00 | 17·45 | 20·60 |
| Elongation, % on 1 in. | 9·0 | 5·0 | 2·0 |
| Rockwell hardness | H 92 | B 49 | B 59 |
| 20 Cr/80 Ni | | | |
| Sintered density, g./c.c. | 7·09 | 7·12 | 6·87 |
| Tensile strength, p.s.i. | 31,300 | 36,200 | 33,000 |
| t.s.i. | 13·95 | 16·15 | 14·75 |
| Elongation, % on 1 in. | 7·8 | 7·3 | 2·4 |
| Rockwell hardness | B 25 | B 42 | B 46 |

**Table II**

**Effect of Sintering Temperature and Time on Properties of Compacts**

Compacting pressure: 50 t.s.i. Sintering conditions: temperature as noted, in hydrogen.

| Powder Composition | Electrolytic Iron Powder + 1% graphite + 10% Cr-Ni Alloy of Composition shown | | | | Electrolytic Iron Powder + 10% of Cr-Ni Alloy of Composition shown | | | |
|---|---|---|---|---|---|---|---|---|
| Sintering temperature, °C. | 1,100 | 1,100 | 1,200 | 1,200 | 1,100 | 1,100 | 1,200 | 1,200 |
| Sintering time, hrs. | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| 80 Cr/20 Ni | | | | | | | | |
| Sintered density, g./c.c. | 6·64 | 6·66 | 6·56 | 6·64 | 6·75 | 6·76 | 6·80 | 6·78 |
| Tensile strength, p.s.i. | 66,100 | 95,300 | 89,000 | 88,100 | 54,800 | 69,400 | 55,200 | 63,500 |
| t.s.i. | 29·50 | 42·55 | 39·75 | 39·35 | 24·45 | 31·00 | 24·65 | 28·35 |
| Elongation, % on 1 in. | 2·0 | 3·0 | 2·3 | 1·8 | 2·5 | 2·8 | 4·3 | 2·5 |
| Rockwell hardness | B 99 | B 95 | B 98 | C 28 | B 54 | B 72 | B 63 | B 69 |
| 50 Cr/50 Ni | | | | | | | | |
| Sintered density, g./c.c. | 6·89 | 6·88 | 6·91 | 6·97 | 6·82 | 6·82 | 6·79 | 6·87 |
| Tensile strength, p.s.i. | 60,300 | 80,400 | 86,500 | 121,000 | 39,100 | 50,800 | 45,700 | 54,400 |
| t.s.i. | 26·90 | 35·90 | 38·60 | 54·05 | 17·45 | 22·70 | 20·40 | 24·30 |
| Elongation, % on 1 in. | 3·7 | 2·5 | 3·7 | 2·5 | 5·0 | 5·2 | 3·7 | 2·5 |
| Rockwell hardness | B 74 | B 89 | B 87 | B 99 | B 49 | B 66 | B 56 | B 66 |
| 20 Cr/80 Ni | | | | | | | | |
| Sintered density, g./c.c. | 7·11 | 7·14 | 7·10 | 7·10 | 7·12 | 7·09 | 7·15 | 7·01 |
| Tensile strength, p.s.i. | 72,200 | 68,600 | 78,800 | 82,200 | 36,200 | 36,900 | 34,700 | 37,700 |
| t.s.i. | 32·25 | 31·05 | 35·20 | 36·70 | 16·15 | 16·45 | 15·50 | 16·85 |
| Elongation, % on 1 in. | 2·5 | 4·3 | 3·2 | 3·6 | 7·3 | 8·0 | 9·0 | 5·4 |
| Rockwell hardness | B 83 | B 84 | B 90 | B 93 | B 42 | B 31 | B 29 | B 45 |

**Table III**

**Effects of Heat-treatment on Properties of Electrolytic Iron Powder + Alloys of varied Chromium-Nickel Contents + 1% Graphite**

| Powder Composition | Electrolytic Iron + 1% Graphite + 10% Cr 80—Ni 20 Alloy | | | | Electrolytic Iron + 1% Graphite + 10% Cr 50—Ni 50 Alloy | | | | Electrolytic Iron + 1% Graphite + 10% Cr 20—Ni 80 Alloy | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Condition of Compact | Tensile Strength | | Elongn. % on 1 in. | Rock-well Hard-ness | Tensile Strength | | Elongn. % on 1 in. | Rock-well Hard-ness | Tensile Strength | | Elongn. % on 1 in. | Rock-well Hard-ness |
| | p.s.i. | t.s.i. | | | p.s.i. | t.s.i. | | | p.s.i. | t.s.i. | | |
| Sintered .. | 66,100 | 29·50 | 2·0 | B 99 | 60,300 | 26·90 | 3·7 | B 74 | 72,200 | 32·25 | 2·5 | B 83 |
| Coined .. | 63,000 | 28·15 | 2·0 | B 95 | 76,200 | 34·00 | 3·5 | B 88 | 76,300 | 34·05 | 4·6 | B 85 |
| Coined and annealed .. | 83,000 | 37·05 | 2·0 | B 95 | 76,300 | 34·05 | 3·5 | B 88 | 71,000 | 31·70 | 3·5 | B 84 |
| Coined and quenched .. | 84,400 | 37·70 | 2·0 | B 100 | 87,500 | 39·05 | 1·2 | C 30 | 76,900 | 34·35 | 1·7 | C 25 |
| Coined, quenched and annealed .. | 92,900 | 41·45 | 2·0 | B 96 | 89,200 | 39·80 | 1·1 | B 100 | 99,500 | 44·40 | 0·5 | C 15 |
| Quenched .. | 84,000 | 37·50 | 2·0 | B 97 | 90,300 | 40·30 | 1·2 | B 101 | 75,800 | 33·85 | 2·3 | C 19 |
| Quenched and annealed .. | 77,300 | 34·50 | 2·0 | B 95 | 78,600 | 35·10 | 1·2 | B 94 | 88,600 | 39·55 | 2·3 | C 16 |

The effect of sintering conditions, on the properties of ternary mixtures containing 10 per cent. of the chromium-nickel alloy powder, and of graphite-containing compacts, was also studied: results are given in table II. From these data it will be observed that an appreciable increase in tensile strength was achieved by lengthening the sintering time at 1100°C. from one to two hours, or by raising the sintering temperature to 1200°C. for the one-hour treatment. Hardness and elongation values remained substantially constant for all conditions of treatment. With the compacts containing 50-50 alloy powder increase in temperature of sintering and lengthening of time of treatment had a cumulative effect. Tensile strength after sintering for 2 hours at 1200°C. was double that produced by one hour at 1100°C. In the compacts containing 20-80 chromium-nickel powder the influence of increase in time and temperature of sintering was much less pronounced. It will be noted that, in general, the presence of 1 per cent. of graphite confers appreciable increase in tensile strength and hardness compared with the properties of graphite-free alloy.

Various forms of heat-treatment were applied to compacts containing electrolytic iron and 1 per cent. of graphite with 10 per cent. of alloy powder of the three compositions. The results, shown in table III, indicate that high tensile strength was obtained on specimens coined, quenched and annealed, and that this rise was accompanied by increase in hardness. No appreciable change in elongation values was observed. The results suggest that this is a promising line of approach as a means of increasing the strength of powder-made materials.

**Powder-Metallurgy Symposium**

See abstract on p. 126.

**Sprayed Stainless-Steel Coatings**

'Stainless Steel Spraying of Drums and Shafts in the Chemical and Food Industries.' *Electroplating and Metal Spraying*, 1953, vol. 6, July, pp. 267, 269-71.

Short, practical discussion of the sphere of application of sprayed stainless-steel coatings in chemical-engineering and other engineering industries. Examples of successful use quoted in the article include spraying of a margarine-freezing drum with stainless steel; patching, *in situ*, of a large paper-mill cylinder which became etched in parts due to a combination of wear and corrosion; spraying of a 15-ft. hydraulic ram, in the plant of a rubber manufacturer.

It is emphasized that although sprayed coatings of stainless steel can be used with outstanding success in suitable applications, it is imperative that neither this nor any other material should be recommended for applications in the chemical or food industries unless its suitability has been unquestionably established, either from previous experience or from results of tests under service conditions, on samples of the material under consideration.

**Sea-Water Tests on Beryllium-Copper, Cupro-Nickel and Other Alloys**

J. T. RICHARDS: 'The Corrosion of Beryllium-Copper Strip in Sea Water and Marine Atmospheres.' *Amer. Soc. Testing Materials*, Preprint 71, June, 1953; 18 pp.

The author reports results of corrosion tests on beryllium-copper and beryllium-cobalt-copper, carried out at Harbor Island and at Kure Beach, N.C. Strip specimens were exposed to quiet and to flowing sea water, rotation through the water at high velocity, jet impingement, and galvanic action. The materials were

also subjected to marine atmosphere at 80 and 800 ft. from the sea. In addition, the effects of time, velocity, temper, heat-treatment, and edge condition were investigated.

Phosphorus-deoxidized copper, arsenical Admiralty metal, iron-containing 70-30 cupro-nickel, and phosphor-bronze were exposed for comparative purposes. Results are expressed in terms of weight-loss and deterioration in strength and elongation.

The results indicate that the corrosion-resistance of beryllium-copper, although inferior to that of cupro-nickel, compares favourably with that of copper under many conditions of sea-water or marine-atmosphere exposure, and that it can therefore be considered for marine applications requiring high strength coupled with the corrosion-resistance of copper.

### Corrosion-Resistance of High-Strength Low-Alloy Steels: Correlated Data

C. P. LARRABEE: 'Corrosion-Resistance of High-Strength Low-Alloy Steels as Influenced by Composition and Environment.' *Corrosion*, 1953, vol. 9, Aug., pp. 259-71.

This paper comprises a critical evaluation of experimental data culled from a wide range of sources, on the behaviour of high-tensile low-alloy steels exposed in various atmospheres, in natural water (fresh and salt), and in soil. The resistance of such steels to attack is compared with that of structural carbon and copper steels in the same environments. The high-tensile low-alloy steels considered include a number in which small percentages of nickel are present.

The general major conclusion is that the effects of differences in environment are of such importance (by comparison with variation in composition) that estimation of behaviour in one location cannot safely be made from a knowledge of the resistance of a given steel to corrosion in another location.

The atmospheric corrosion-resistance of some of the high-tensile steels is markedly superior to that of structural carbon and copper steels. The comparative resistance of the individual grades of steel in any one environment is determined by chemical composition: the superiority of one steel over another is believed to be due to differences in the protective qualities of the rust which forms on the exposed surfaces. On sheltered surfaces the high-strength low-alloy types, although still somewhat more resistant, do not show so great a degree of difference from the structural steels as in unsheltered conditions.

In most natural waters, and in soils, the presence of small amounts of alloying elements in the steel does not play a major part in determining the average corrosion rates, but steels containing 3-4 per cent. of alloys do not pit so severely as structural carbon steels.

### Influence of Composition of Steel on Resistance of Organic Coatings to Atmospheric Corrosion

F. L. LAQUE and J. A. BOYLAN: 'Effect of Composition of Steel on the Performance of Organic Coatings in Atmospheric Exposure.' *Corrosion*, 1953, vol. 9, July, pp. 237-41.

The investigation reported in this paper was undertaken to determine the possibility of reducing corrosion of automobile bodies by the use of steels containing alloying elements. The tests made were designed to ascertain to what extent it would be advantageous to use such steels as bases for the paint coatings usually employed in the automobile industry. The materials tested as bases were: open-hearth iron of exceptionally low copper content, known to be vulnerable to corrosion attack; a representative low-copper steel (0·2 per cent. Cu); a commercial low-alloy high-strength steel of the nickel-copper type; a commercial low-alloy high-strength steel containing small amounts of chromium, nickel, silicon, copper and phosphorus; an experimental steel containing a total of about 0·5 per cent. nickel plus copper; a steel supposed to be representative of standard automobile body stock, but found to contain unusually high copper (0·20 per cent., as compared with normal residual copper of 0·08 per cent. in this type of steel).

A common automobile paint cycle was used, with and without zinc phosphate (Bonderite) pre-treatment. Test panels, bare and coated, were exposed at positions 80 and 800 ft. from the sea at Kure Beach, N.C., in an industrial atmosphere at Bayonne, N.J., and in a rural atmosphere at Morenci, Mich.

The principal conclusions are:

The low-alloy chromium - nickel - silicon - copper - phosphorus steel gave the best resistance, with copper steel ranking next. The low-copper open-hearth iron gave the lowest resistance, both painted and bare.

Zinc-phosphate pre-treatment improved the performance of all the painted steels: the advantage so conferred increased with the severity of the atmospheric conditions and the susceptibility of the steels to corrosion; the best performance in the tests was given by the low-alloy complex steel, pre-treated by zinc-phosphate.

Exposure of only a few weeks 80 ft. from the sea gave the same rating as that obtained after much longer exposure in the other atmospheres. Marine atmosphere 800 ft. from the sea was less damaging to bare steels, but more damaging to painted steels, than the industrial atmosphere.

**Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks.***

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